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ARIES



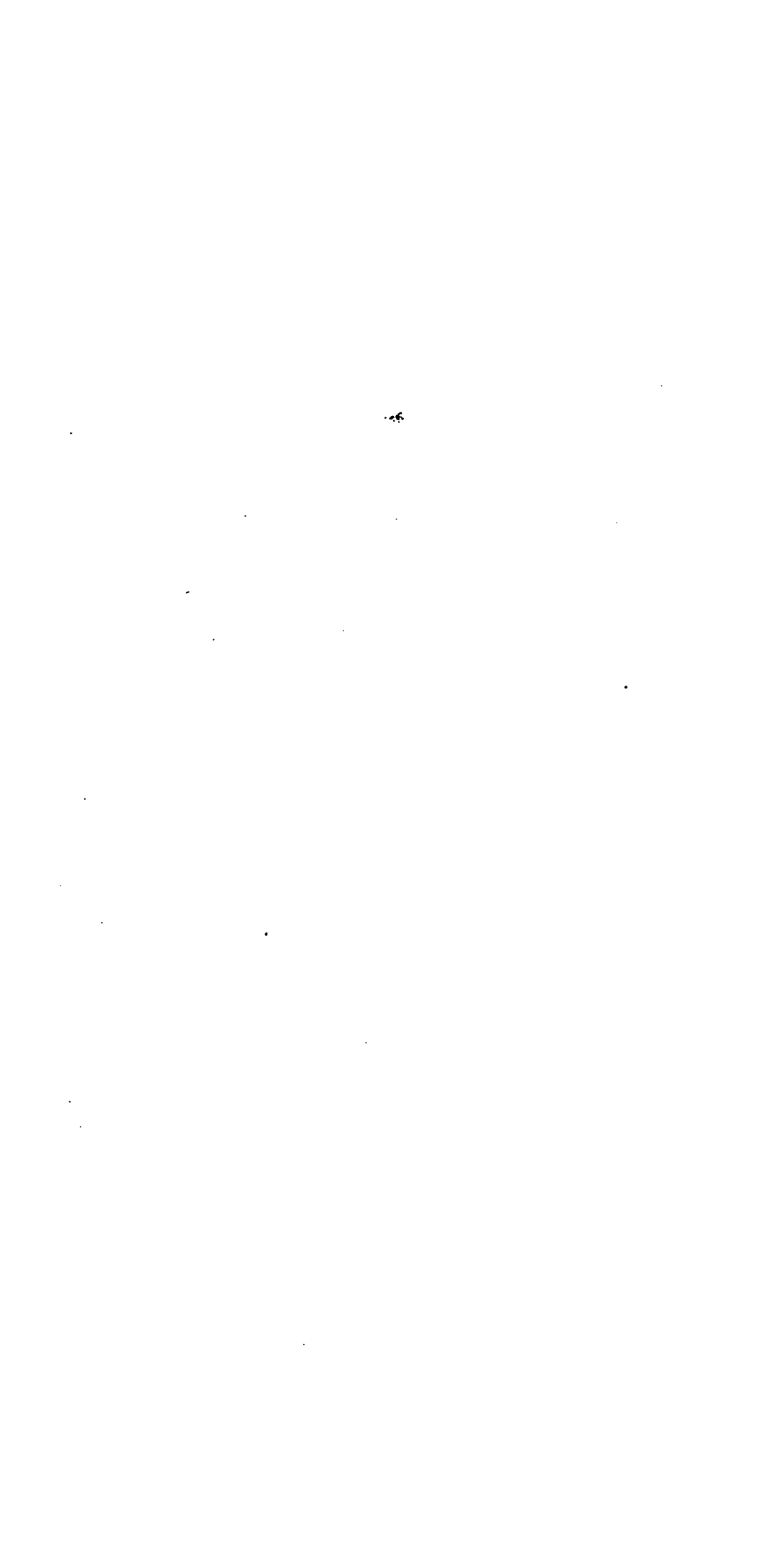
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STANDARDS AND TESTS
FOR
REAGENT CHEMICALS



**STANDARDS AND TESTS
FOR
REAGENT CHEMICALS**

BY

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PREFACE

The large number of new chemical and allied industries that have sprung up in our country, and the recent extensive expansion of the old, have greatly increased the consumption of reagent chemicals. Research, analytical, and control chemistry have entered upon new and very broad fields of activity as a result of the impulse acquired by manufacturing. New reagent chemicals, as well as more complete and more appropriate lines of earlier reagents, are needed to assist in the vital problem of production, or to suitably control the quality of the factory output. But the very chemicals by means of which this is done must themselves be tested and standardized. In other words, our chemical tools need calibration. Certain desired degrees of purity in our laboratory chemicals must be insured.

Recognizing the above condition this text has been prepared. It describes and standardizes the more important testing chemicals used today in factory and other laboratories, particularly those produced successfully on a commercial scale. A few, although very important, are not described, as they are generally prepared extemporaneously.

Rather full descriptions of the physical properties of chemicals, action of light and air, precautions to be observed in storing, statement of uses, etc., are made. In many cases, too, the physical constants of reagent chemicals produced in a commercial way, are, for purposes of information, compared with those obtaining on laboratory specimens, doubtless of extreme purity. It is hoped that certain readers, especially younger men, or those without convenient reference works at hand may find these somewhat elementary features of the text of value.

The statements of the "Maximum Limits of Impurities," so often occurring throughout the book, are not the amounts of impurities present, but are the maxima permissible in

chemicals suitable for miscellaneous reagent purposes. The reagents of the market are frequently, if not generally, well below these maxima.

The percentage figures in which the above limits are stated were in part determined by the customary quantitative tests, and to this extent are accurate; in part by qualitative tests in which the reactions in unknowns are compared with those in knowns. Such comparative tests have a real value in determining the usefulness of reagents, although they should not be regarded as exact. The tests will insure good reagents even though the stated percentages of impurities may not be entirely correct.

The intimate personal knowledge of fine chemicals, gained from many years experience in charge of the control laboratories of Merck & Co. has furnished the author the foundation and the justification for undertaking to prepare such a text as is here presented.

B. L. MURRAY.

RAHWAY, N. J., May, 1919.

EXPLANATORY NOTE

Certain features and expressions frequently met with in the text require a word of explanation for their better understanding. The following subjects deserve notice:

Mils.—The word “mil” is used as a short form of the word “milliliter,” the thousandth part of a liter, heretofore commonly but erroneously termed “cubic centimeter” or “c.c.”

Atomic Weights.—The International Atomic Weights for 1918, $O = 16$, are used as a basis for quantitative calculations, factors, etc. The table is to be found on page 372.

Specific Gravities.—Unless otherwise stated, these are taken at $+15^{\circ}$ C. compared with water at the same temperature.

Solubilities stated in the descriptions are not intended as standards, but are inserted for information and are closely approximate only. They are based on published solubilities at 15° C. Sometimes these approximate solubilities are omitted when no authoritative figures are available.

Parts by weight are to be understood when the word “parts” is employed, as for instance in stating solubilities.

Specific Gravities, Boiling Points and Melting Points frequently given in the descriptions, refer to the reagent grade of the chemical produced on a commercial scale. Parenthetical phrases generally follow—*e. g.* “(theory 1.289 at $15^{\circ}/0^{\circ}$ C.)”—giving the corresponding figures as recorded in literature, presumably for the purest specimens produced experimentally in the laboratory; these have been inserted for information and comparison. The parenthetical data have been taken from Van Nostrand’s Chemical Annual, 1918, edited by Dr. John C. Olsen; or, when not found there, from other authoritative sources.

Volumetric Determinations may be made with other standard solutions than those directed in the text provided the substitute is adapted to the case.

Time Limits of Reactions.—When no time is specified for observing the result of a test, at least one minute is to be allowed for the reaction to take place.

Abbreviations of the names of periodicals to which reference is made are those ordinarily used in English language abstract journals.

REAGENTS, TESTS AND TEST SOLUTIONS

Alcohol of 95% strength is to be understood by the term "alcohol."

Ammonia Water.—Unless otherwise specified, ammonia water, 10%, is to be used.

Sulphuric Acid.—When not otherwise specified the acid of specific gravity 1.84 is to be used.

Nitric Acid.—When not otherwise specified in the text, nitric acid of specific gravity 1.20 is to be used.

Hydrochloric Acid.—Unless otherwise specified, the acid of specific gravity 1.18 to 1.19 is to be used when hydrochloric acid is directed in making the tests.

Stannous Chloride Solution.—The solution described on page 356 is to be used.

Ammonium Carbonate Solution.—Dissolve 5 gm. of ammonium carbonate in a mixture of 5 mils of ammonia water and 15 mils of water.

Diphenylamine Solution.—Dissolve 0.5 gm. of diphenylamine in a mixture of 100 mils of sulphuric acid, sp. gr. 1.84, and 20 mils of water.

Ammoniacal Silver Nitrate Solution.—Dissolve 1 gm. of silver nitrate in 20 mils of water, add ammonia water by drops, agitating the mixture, until the precipitate first formed is almost but not entirely redissolved, and filter.

Alcoholic Ammoniacal Silver Nitrate Solution.—Dissolve 1 gm. of silver nitrate in 100 mls of 95% alcohol and add 5 mls of alcoholic solution of ammonia containing 9% to 10% of NH_3 .

Ammoniacal Lead Acetate Solution.—To the ordinary 10% test solution of lead acetate add sufficient ammonia water to impart a distinctly ammoniacal odor. If necessary filter to remove any precipitate of lead carbonate which may form.

Indigo Solution.—Dissolve 0.1 gm. of synthetic indigo, described on page 170, in 2 mls of fuming sulphuric acid, containing about 15% of free SO_3 , and dilute with water to 100 mls.

Metaphenylenediamine Hydrochloride Solution.—Dissolve 1 gm. of metaphenylenediamine hydrochloride in 100 mls of water. If colored, the solution is to be decolorized by heating with previously ignited animal charcoal and filtering.

Acid Ferrous Sulphate Solution.—Dissolve 3 gm. of clear crystals of ferrous sulphate in 90 mls of water which has been previously boiled to expel air, and add sufficient sulphuric acid, sp. gr. 1.84, to make 100 mls. This solution should be prepared immediately before use.

Acid Manganese Sulphate Solution.—Dissolve 10 gm. of manganese sulphate in 50 mls of water and add sufficient 10% sulphuric acid to make 100 mls.

Methyl Alcohol-Hydrochloric Acid.—Pass thoroughly dried hydrochloric acid gas into anhydrous methyl alcohol until saturated, maintaining a low temperature during the process. The hydrochloric acid gas may be produced readily by dropping concentrated sulphuric acid into concentrated hydrochloric acid. The reagent does not keep well and should be reasonably fresh when used.

Marsh Test for Arsenic.—The Marsh apparatus referred to in the text consists of a generating flask of about

200 mils capacity, through the stopper of which passes a thistle-tube for the introduction of acid and liquids to be tested, and an outlet tube leading to a drying-tube which at its other end connects with the reduction tube. The drying tube is filled partly with dry, granular calcium chloride, partly with absorbent cotton which has been saturated with lead acetate solution and afterwards dried. Start the operation with 20 gm. of arsenic-free zinc and sufficient 20% sulphuric acid to obtain a steady but not too copious flow of gas. If the zinc is not sufficiently active, introduce a solution of 4 gm. of arsenic-free cadmium sulphate in 8 to 10 mils of water and then, when the air is all expelled, the solution to be tested.

Test solutions mentioned without qualification in the text and not described above are the usual laboratory solutions.

STANDARDS AND TESTS FOR REAGENT CHEMICALS

ACETONE

(Dimethyl-ketone)

$(\text{CH}_3)_2\text{CO}$

Mol. Wt. 58.06

Acetone is a clear, colorless liquid with a characteristic ethereal odor. It is miscible in all proportions with water, alcohol, ether and many other liquids. It has a specific gravity of about 0.798 (theory 0.797 at $15^\circ/4^\circ$ C.), boils at about 57° C. (theory 56.53° C.) and contains at least 99% of $(\text{CH}_3)_2\text{CO}$. It is neutral to litmus, somewhat hygroscopic and highly inflammable.

In pure condition it is not easily affected by light, but acetone containing impurities, such as usually occur in the commercial article, when exposed to light often becomes yellow and acquires acidity and a high content of substances reducing permanganate.

Acetone is used chiefly as a solvent for fats, resins, nitro-celluloses, etc.; in the analysis of rubber and asphalt; as a precipitant for albumin; and in photography. It should be kept in air-tight containers in a cool place and should be stored and handled away from lights and fires in order to avoid ignition of the acetone or its vapor.

Maximum Limits of Impurities

Residue on Evaporation	0.0025%
Acids and Alkalis	0.0000%
Aldehydes	0.0000%
Substances Insoluble in Water	0.0000%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Residue on Evaporation.—Evaporate 50 mls of acetone at the temperature of the steam-bath. Not over 0.001 gm. of residue should remain.

Aldehydes.—Warm 10 mls of acetone with 5 mls of ammoniacal silver nitrate solution for fifteen minutes at about 50° C. in a place protected from light. No brown color or deposit of metallic silver should be produced in the mixture.

Substances Insoluble in Water; Acids and Alkalies.—Mix 10 mls of acetone with 10 mls of water. The mixture should be clear and neutral to litmus paper.

Substances Reducing Permanganate.—Add 0.05 ml of tenth-normal solution of potassium permanganate to 10 mls of acetone and let the mixture stand fifteen minutes, at a temperature of not over 15° C. The pink color produced by the addition of the permanganate should not be entirely discharged at the end of that time.

Quantitative Method.—Dilute a sample of acetone to a convenient volume, add an aliquot containing approximately 0.02 gm. of the acetone to 25 mls of normal solution of potassium hydroxide and then add with constant agitation of the mixture about 35 mls of tenth-normal iodine solution. Allow the mixture to stand fifteen minutes, then add 26 mls of normal solution of hydrochloric acid and at once titrate the residual iodine with tenth-normal solution of sodium thiosulphate, using starch as indicator. Conduct another test with the same quantities of reagents but without the acetone and subtract the quantity of tenth-normal iodine solution consumed in this blank test from that consumed in the test with the acetone. At least 99% of $(\text{CH}_3)_2\text{CO}$ should be found.

1 ml of tenth-normal $\text{I} = 0.00096771$ gm. of $(\text{CH}_3)_2\text{CO}$,
log 98574.

ACID ACETIC, 99.5%**(Glacial Acetic Acid)**

Mol. Wt. 60.04

Acetic acid, 99.5%, is a clear, colorless liquid having a pungent odor. It is clearly miscible with water or alcohol. The acid has a specific gravity of about 1.048 at 25° C., congeals at a temperature not lower than 15.6° C., and contains at least 99.5% of $\text{HC}_2\text{H}_3\text{O}_2$. Its vapor is inflammable.

Acetic acid has occasionally been found to contain traces of copper, which though not detected by the ordinary tests for heavy metals, impart a bluish tint to the acid, visible when a large volume is inspected against a white background.

Besides being a general neutralizing and acidifying agent acetic acid is used principally as a solvent for essential oils, resins, etc.; as a vehicle for the application of other reagents, such as chromic acid and iodine chloride or bromide; for the detection of rosin oil in linseed oil; the precipitation of albumin; and in microscopy.

Glacial acetic acid should be stored in glass-stoppered bottles and kept at a temperature above its congealing point in order to avoid difficulty and danger of loss by breakage attendant upon melting the congealed acid. The containers are best sealed with an insoluble or inert substance, such as plaster paris or paraffin. Resins and waxes, if used, would be likely to be dissolved to the detriment of the acid. When properly stored it will keep indefinitely.

Maximum Limits of Impurities

Non-volatile Matter	0.0005%
Chlorides (Cl)	0.0003%
Sulphates (SO_4)	0.0003%
Sulphurous Acid (SO_2)	0.0200%
Formic Acid (HCOOH)	0.1000%
Iron (Fe)	0.0001%
Other Heavy Metals	0.0000%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 100 mils of acetic acid to dryness and ignite the residue. Not more than 0.0005 gm. should be left.

Chlorides.—Dilute 5 mils of acetic acid with 50 mils of water and add 5 mils of nitric acid and some silver nitrate solution. The mixture should not appear changed.

Sulphates.—Add a few milligrams of sodium carbonate to 50 mils of the acid, evaporate to dryness, dissolve the residue in 50 mils of water, add barium chloride solution and let stand twelve to eighteen hours. Any precipitate forming within this time should not weigh more than 0.0005 gm.

Sulphurous and Formic Acids.—Neutralize 5 mils of the acid with ammonia water, avoiding an excess, add 2 to 3 mils of silver nitrate solution and boil the mixture two minutes. No dark color or deposit should be produced.

Iron.—Dilute 10 mils of the acid with 10 mils of water, add a few drops of nitric acid and boil the mixture. Then add 5 mils of potassium sulphocyanate solution. No red color should develop.

Other Heavy Metals.—Dilute 20 mils with 100 mils of water and pass in hydrogen sulphide gas. No color or precipitate should be produced.

Supersaturate 20 mils of the acid with a slight excess of ammonia water, dilute the mixture to 100 mils and add a few drops of ammonium sulphide solution. No dark color should be produced.

Substances Reducing Permanganate.—Dilute 2 mils of the acid with 10 mils of water, add 0.1 mil of tenth-normal potassium permanganate solution and allow to stand two hours at a temperature not exceeding 25° C. The pink color should not be entirely changed to brown.

Quantitative Method.—The strength of the glacial acid may easily be ascertained by determining its congealing

point* or specific gravity and referring to acetic acid tables;† or if desired the acid may, after suitable dilution, be titrated with standard alkali, using phenolphthalein as indicator. Not less than 99.5% of $\text{HC}_2\text{H}_3\text{O}_2$ should be found.

1 mil of normal $\text{NaOH} = 0.06004$ gm. of $\text{HC}_2\text{H}_3\text{O}_2$,
log 77844.

ACID ACETIC, 36%

$\text{HC}_2\text{H}_3\text{O}_2$,

Mol. Wt. 60.04

Acetic acid, 36%, is a clear colorless liquid of specific gravity about 1.049 and contains 35 to 37% of $\text{HC}_2\text{H}_3\text{O}_2$.

The 36% acid is commonly used as an acidifying agent in cases where the use of the glacial acid is unnecessary or inadvisable. The official test solution of the U. S. Pharmacopœia, 9th Revision, is 36%. This acid is stored preferably in glass-stoppered containers.

Maximum Limits of Impurities

Non-volatile Matter	0.0002%
Chlorides (Cl)	0.0001%
Sulphates (SO_4)	0.0001%
Sulphurous Acid (SO_2)	0.0080%
Formic Acid (HCOOH)	0.0400%
Iron (Fe)	0.0001%
Other Heavy Metals	0.0000%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 100 mls of the acid and ignite the residue. Not more than 0.0002 gm. should be left.

Chlorides.—Dilute 12.5 mls with 40 mls of water, add 5 mls of nitric acid and some silver nitrate solution. The mixture should not appear changed.

* A method of determining the congealing point of liquids is official in the U. S. Pharmacopœia, 9th Revision, page 599.

† A congealing point table may be found on page 374. For specific gravity tables consult Van Nostrand's Chemical Annual, 1918, or the U. S. Pharmacopœia, 9th Revision.

Sulphates.—Add a few milligrams of sodium carbonate to 100 mls of the acid, evaporate to dryness, dissolve the residue in 50 mls of water, add barium chloride solution and allow to stand twelve to eighteen hours. Any precipitate forming in this time should not weigh more than 0.0003 gm.

Sulphurous and Formic Acids.—Neutralize 12.5 mls of the acid with ammonia water, avoiding an excess, add 2 to 3 mls of silver nitrate solution and boil the mixture two minutes. No dark color or deposit should be produced in the liquid by this treatment, although occasionally a faint yellowish or brownish tint may be observed.

Iron.—To 20 mls add a few drops of nitric acid, boil and then add 5 mls of potassium sulphocyanate solution. No red color should develop.

Heavy Metals.—Dilute 50 mls with 60 to 70 mls of water and pass in hydrogen sulphide gas. No color or precipitate should be produced.

Supersaturate 50 mls of the acid with a slight excess of ammonia water, dilute the mixture to 100 mls and add a few drops of ammonium sulphide solution. No dark color should be produced.

Substances Reducing Permanganate.—Dilute 5 mls of the acid with 5 mls of water, add 0.1 ml of tenth-normal potassium permanganate solution and allow to stand two hours at a temperature not exceeding 25° C. The pink color imparted by the permanganate should not be entirely changed to brown at the end of this time.

Quantitative Method.—Titrate the acid, suitably diluted, with standard alkali solution, using phenolphthalein as indicator. At least 35% and not more than 37% of $\text{HC}_2\text{H}_3\text{O}_2$ should be found.

1 mil of normal $\text{NaOH} = 0.06004$ gm. of $\text{HC}_2\text{H}_3\text{O}_2$,
log 77844.

**ACID ACETIC, CONFORMING TO THE DICHR-
MATE TEST** $\text{HC}_2\text{H}_3\text{O}_2$

Mol. Wt. 60.03

Acetic acid, conforming to the dichromate test, has the appearance and general properties of Acid Acetic, 99.5%. It congeals at a temperature not lower than $+12.8^\circ \text{C}$. and contains at least 98% of $\text{HC}_2\text{H}_3\text{O}_2$.

The acid is particularly free from reducing substances and is employed chiefly in preparing iodine solutions used in the analysis of fats, oils, resins, etc.; in determining colophony in shellac and turpentine, and rosin oil in linseed oil.

The reagent should be stored with the same precautions as Acid Acetic, 99.5%.

Maximum Limit of Impurity

Substances Reducing Dichromate 0.0000%

Methods of Testing

Substances Reducing Dichromate.—Mix 10 mls of the acid with 10 mls of sulphuric acid, containing in solution 0.02 gm. of potassium dichromate, and allow the mixture to stand half an hour at 15° to 20°C . No green color should develop in the liquid.

Quantitative Method.—Determine the strength of the acid as directed under Acid Acetic, 99.5%. At least 98% of $\text{HC}_2\text{H}_3\text{O}_2$ should be found.

(ACID) ACETIC ANHYDRIDE $(\text{CH}_3\text{CO})_2\text{O}$

Mol. Wt. 102.07

Acetic anhydride is a colorless liquid having a pungent odor. It boils at about 137°C . (theory 137.9°C .) and has a specific gravity of about 1.08. It is readily soluble in alcohol, ether or chloroform, but slowly soluble in water with which it forms acetic acid. The reagent contains at least 98.5% of $(\text{CH}_3\text{CO})_2\text{O}$.

The yellow color frequently observed in acetic anhydride, especially in the commercial article, is difficult to remove by simple distillation, but a colorless product may be obtained by distilling it from a little phosphoric anhydride.

The acetic anhydride of the market contains more or less acetic acid and the method of direct titration as given under the methods of testing yields the percentage of total acetic anhydride, including that existing as acetic acid. Assuming that the article consists only of a mixture of acetic anhydride and acetic acid, the percentage of each may be calculated from the result of the direct titration or may be learned by referring to the table on page 373. Other methods of determining the actual content of acetic anhydride are in use. For a résumé of some of these methods and their comparative value see *The Examination of Acetic Anhydride*, Radcliffe and Medofski, *J. Soc. Chem. Ind.*, **36**, 628 (1917); *abst. Chem. Abst.*, **11**, 2649 (1917).

Acetic anhydride is mainly used in the examination of wool fat, glycerin, fatty and volatile oils, resins, etc.; in acetylations; and in organic analysis and synthesis.

Acetic anhydride should be stored in glass-stoppered containers and should be protected from contact with air and moisture. For sealing the stoppers paraffin or plaster paris is to be preferred. Sealing-wax or other resinous substance liable to contaminate the reagent should not be used.

Maximum Limits of Impurities

Non-volatile Matter	0.0025%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0025%
Sulphates (SO ₄)	0.0010%
Other Sulphur Compounds (S)	0.0032%
Phosphorus Compounds (P)	0.0017%

Methods of Testing

Non-volatile Matter.—Evaporate 20 mls of acetic anhydride, dry the residue at 100° C. and weigh it. It should not weigh more than 0.0005 gm.

Heavy Metals.—Dissolve 1 mil in 20 mils of water and add hydrogen sulphide water. No color or turbidity should be visible even after adding ammonia water in slight excess.

Chlorides.—Dissolve 2 mils of acetic anhydride in 20 mils of water, add 2 mils of nitric acid and some silver nitrate solution. Not more than a slight opalescence should develop.

Sulphates.—Dissolve 20 mils in 100 mils of water, add 5 mils of hydrochloric acid and some barium chloride solution and allow to stand about fifteen hours. No precipitate of barium sulphate should form.

Other Sulphur Compounds (Thioacetic Acid).—Dissolve 5 mils of acetic anhydride in 50 mils of water, add 0.1 mil of tenth-normal iodine solution followed by a few drops of starch solution. A blue color should be produced.

Phosphorus Compounds.—Dissolve 5 mils in 20 mils of water, add 10 mils of nitric acid and heat to boiling. Cool, add 25 mils of ammonium molybdate solution and allow the mixture to stand two hours at about 40° C. No yellow precipitate should be produced.

Quantitative Method.—Dissolve the anhydride in water* and titrate it with standard alkali solution, using phenolphthalein as indicator. Not less than 98.5% of $(\text{CH}_3\text{CO})_2\text{O}$ should be found.

1 mil of normal NaOH = 0.051035 gm. of $(\text{CH}_3\text{CO})_2\text{O}$,
log 70787.

ACID BENZOIC

$\text{C}_6\text{H}_5\text{COOH}$

Mol. Wt. 122.08

Benzoic acid usually occurs as colorless, glistening needles or scales, soluble in about 300 parts of water and in about 2

*Care should be taken that the anhydride is completely hydrolyzed. This can be accomplished by allowing the solution to stand several hours before titration or by warming gently under reflux condenser for fifteen or twenty minutes.

parts of alcohol or ether; also readily soluble in chloroform or benzene. It melts between 121° and 122° C. (theory 121.25° C. corr.) and contains at least 99.9% of C_6H_5COOH .

To facilitate the handling of this reagent it is sometimes formed into fused pieces. This is done by heating the acid, contained in a covered platinum dish, in an oven at 140° C. until melted, when it is poured into test-tubes. The sticks so obtained are broken up into pieces of convenient size for use. This form has the advantage of greatly diminished bulk and surface exposure and can be used without preliminary drying as the acid is not hygroscopic.

The reagent is intended especially for use as a standard for preparing volumetric solutions of the alkalis; it may also be employed as a calorimetric standard.

The acid should be stored in well-stoppered bottles and protected from the light.

Maximum Limits of Impurities

Inorganic Substances	0.0250%
Chlorine Compounds (Cl)	0.0010%
Organic Impurities	0.0000%
Heavy Metals	0.0000%

Methods of Testing

Inorganic Substances.—Ignite 2 gm. of the acid and weigh the residue. The weight should not exceed 0.0005 gm.

Chlorine Compounds.—Mix 2 gm. of the acid with 0.5 gm. of calcium carbonate (free from chloride), moisten the mixture with a little water, dry and ignite it at a low red heat. Dissolve the residue in a little nitric acid, dilute with water to 10 mls, filter and to the filtrate add silver nitrate solution. Not more than a very faint opalescence should result.

Organic Impurities, Heavy Metals.—Dissolve 1 gm. in a mixture of 10 mls of ammonia water and 10 mls of water. The solution should be clear and colorless. To this solution add hydrogen sulphide water. No change should be apparent.

Quantitative Method.—Dry the acid to constant weight in a desiccator over sulphuric acid, dissolve about 0.5 gm. of the dried acid, accurately weighed, in 30 mls of alcohol and titrate the solution with tenth-normal barium hydroxide solution, using phenolphthalein as indicator. Correct the titration for the amount of the barium hydroxide solution required to neutralize 30 mls of the alcohol. At least 99.9% of C_6H_5COOH should be found.

1 mil of tenth-normal $Ba(OH)_2 = 0.012208$ gm. of C_6H_5COOH , log 08665.

ACID BORIC

(Boracic Acid)

H_3BO_3

Mol. Wt. 62.02

Boric acid for reagent purposes is usually in the form of colorless, transparent crystals, soluble in about 18 parts of water or alcohol and in about 5 parts of glycerin. It is used mainly to detect curcuma and acetanilid and in the determination of ammonia.

Maximum Limits of Impurities

Insoluble Substances	0.0000%
Chlorides (Cl)	0.0020%
Sulphates (SO_4)	0.0100%
Calcium (Ca)	0.0040%
Heavy Metals	0.0000%

Methods of Testing

Insoluble Substances.—Dissolve 1 gm. of boric acid in 10 mls of boiling alcohol. No insoluble residue should be left.

Chlorides.—Dissolve 5 gm. in 100 mls of water and add 5 mls of nitric acid and some silver nitrate solution. No more than a slight opalescence should develop.

Sulphates.—Dissolve 5 gm. in 100 mls of water and add 5 mls of hydrochloric acid and some barium chloride

solution. No change in the appearance of the liquid should be visible within fifteen minutes.

Calcium.—Dissolve 5 gm. in 100 mls of water, render the solution alkaline with ammonia water and add ammonium oxalate solution. The mixture should be clear.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add 10 mls of hydrogen sulphide water. The liquid should be clear and colorless. Then add 5 mls of ammonia water. No dark color or precipitate should be produced.

(ACID) BORIC ANHYDRIDE

(Boron Trioxide; Boric Acid, Fused)

B_2O_3

Mol. Wt. 70.0

Boric anhydride usually occurs as brittle, vitreous, hygroscopic lumps. It is soluble in about 35 parts of water, forming boric acid; also soluble in alcohol. The reagent is especially intended for use in the analysis of silicates. Regarding this use see The Analysis of Silicate and Carbonate Rocks, U. S. Geological Survey, Bull. 422, 86 (1910).

Boric anhydride should be stored in well-stoppered bottles.

Maximum Limit of Impurities

Silica, Alkalis, etc. 0.0100%

Method of Testing

Silica, Alkalis, etc.—Powder 5 gm. of the boric anhydride in an agate mortar,* place the powder in a weighed platinum dish, add 50 mls of methyl alcohol-hydrochloric acid (see page ix), stir the mixture with a platinum wire until the anhydride is completely dissolved, place the dish on a wire gauze and evaporate the liquid over a small flame. If a residue remains treat it in the same way as before with 25 mls of the methyl alcohol-hydrochloric acid, evaporate to dryness and if necessary treat a third time in order to be

* The use of porcelain or other mortars is apt to introduce impurities, due to abrasion by the hard pieces of boric anhydride.

sure that the residue is not some of the anhydride unvolatilized. Finally ignite the dish gently and weigh. The weight of any residue remaining should not exceed 0.0005 gm.

ACID CARMINIC

 $C_{24}H_{22}O_{12}$

Mol. Wt. 494.29

Carminic acid is the red coloring principle of cochineal and the important constituent of carmine. It is usually in the form of a dark reddish-brown powder, easily soluble in water or alcohol, insoluble in chloroform or benzene. It is used principally in the detection of albumin, as a microscopic stain, and, in 1% aqueous solution, as an indicator.

As an indicator it is serviceable in titrating alkalis, especially ammonia, as in Kjeldahl nitrogen determinations. It is well adapted to the titration of alkaloids, morphine, cocaine, atropine and brucine in particular. The indicator is affected but slightly by carbon dioxide or hydrogen sulphide and hence is of value in estimating carbonates and sulphides. For organic acids it is useless. In titrating with alkali solutions the presence of a little ammonium salt increases the sharpness of the end reaction.

Maximum Limit of Impurity

Insoluble Matter 0.0000%

Methods of Testing

Insoluble Matter.—Dissolve 1 gm. in 2 mls of water. Solution should be complete. Add to the solution 20 mls of alcohol. No turbidity should be produced.

Sensitiveness.—Dissolve 5 gm. of ammonium chloride in 50 mls of water and add 1 drop of 1% aqueous solution of carminic acid. The yellowish-red color of the mixture should change to violet-red on adding 0.05 ml of tenth-normal sodium hydroxide solution.

ACID CITRIC

Mol. Wt. 210.11

Citric acid is usually in the form of colorless crystals, which effloresce when exposed to warm air. It is soluble in less than 1 part of water, in about 1.5 parts of alcohol, and in about 50 parts of ether. It contains at least 99.5% of $\text{H}_3\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$.

Citric Acid is used principally in the determination of citrate-soluble phosphoric acid; in separating iron and aluminum; in differentiating albumin from mucus and testing urine for albumin, mucin, glucose, bile pigments, etc.

The reagent should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Oxalic Acid ($\text{H}_2\text{C}_2\text{O}_4$)	0.1000%
Sulphates (SO_4)	0.0020%
Calcium (Ca)	0.0100%
Lead (Pb)	0.0010%
Organic Impurities	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 5 gm. of citric acid. Not more than 0.0025 gm. of residue should be left.

Oxalic Acid.—Dissolve 10 gm. of the citric acid in 100 mls of water and nearly neutralize with ammonia water. Add 5 mls of calcium chloride solution and allow it to stand twenty-four hours. No precipitate of calcium oxalate should form.

Sulphates.—Dissolve 2 gm. of the acid in 20 mls of water, add barium chloride solution and allow to stand about fifteen hours. No precipitate of barium sulphate should form.

Calcium.—Dissolve 2 gm. in 20 mls of water and add ammonium oxalate solution. The liquid should remain clear.

Lead.—Dissolve 5 gm. of citric acid in 10 mls of water, add 12 mls of ammonia water and about 10 mls of hydro-

gen sulphide water. No dark color should appear in the mixture.

Organic Impurities (Tartaric Acid, Sugar, etc.).—Mix 1 gm. of the powdered acid with 10 mls of sulphuric acid and heat the mixture in a steam-bath for half an hour. At the end of this time the mixture may have acquired a yellowish to brownish-yellow color but no dark-brown or reddish-brown color should have developed.

Quantitative Method.—Titrate an aqueous solution of the acid with standard alkali solution, using phenolphthalein as indicator. At least 99.5% of $\text{H}_3\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$ should be found.

1 mil of normal $\text{NaOH} = 0.070037$ gm. of $\text{H}_3\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$,
log 84533

ACID GALLIC

$\text{C}_7\text{H}_5\text{O}_6 + \text{H}_2\text{O}$

Mol. Wt. 188.10

Gallic acid occurs as white or pale fawn-colored crystals or crystalline powder. It is soluble in about 85 parts of water and in about 6 parts of alcohol; difficultly soluble in ether and almost insoluble in chloroform.

Gallic acid may be distinguished from tannic acid by the fact that the former does not cause a precipitate in a solution of gelatine or starch. When introduced into even very dilute solutions of ferric salts gallic acid produces a bluish-black color or precipitate, but this reaction does not take place with ferrous salts. On account of this behavior the acid is often used to detect small amounts of ferric salts, as for instance those occurring in mineral waters. It is also used for the detection of free mineral acids.

Gallic acid is somewhat sensitive to light and should be protected from undue exposure to it. It should also be kept from contact with ammonia vapors by which it is easily discolored. Well-stoppered, amber-colored bottles are therefore preferable as containers. Solutions of gallic acid do not keep well and should be freshly prepared when required.

Maximum Limits of Impurities

Insoluble Substances	0.0000%
Non-volatile Matter	0.0500%
Sulphates (SO ₃)	0.0050%
Excess Water	0.0000%

Methods of Testing

Insoluble Substances.—Dissolve 1 gm. in 20 mls of hot water. No undissolved residue should remain.

Non-volatile Matter.—Ignite 1 gm. of the acid. Not more than 0.0005 gm. of ash should be left.

Sulphates.—Dissolve 1 gm. in 50 mls of hot water, add 1 mil of hydrochloric acid and some barium chloride solution and let the mixture stand one hour at about 50° C. No precipitate of barium sulphate should be produced.

Excess Water.—Dry the acid to constant weight at 100° C. The loss in weight should not be more than 10%.

ACID HYDRIODIC, Sp. Gr. 1.70

HI

Mol. Wt. 127.93

Hydriodic acid of specific gravity 1.70 is usually a yellowish to brownish liquid, miscible with water. It contains about 57% of HI. When freshly prepared it is colorless but quickly discolors when exposed to the air, iodine being liberated.

The reagent is especially intended for the determination of methoxyl in volatile oils and other substances; it is also used as a reducing agent in organic synthesis. Regarding the methoxyl determination see Allen's Commercial Organic Analysis, 4th ed., IV, 240 (1911).

The acid should be stored in tightly-stoppered bottles and protected as much as possible from contact with the air.

Maximum Limits of Impurities

Residue on Evaporation	0.0400%
Residue on Ignition	0.0100%
Phosphorus	0.0000%
Sulphuric Acid (H ₂ SO ₄)	0.0100%

Methods of Testing

Residue on Evaporation, Residue on Ignition, Phosphorus.—Evaporate 5 gm. of hydriodic acid on the steam-bath and weigh the residue. The weight should not exceed 0.002 gm. Gently ignite this residue and weigh again. Not more than 0.0005 gm. should be left, and during the ignition the residue should not burn.

Sulphuric Acid.—Dilute 3 mls of the acid with 50 mls of water, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Quantitative Method.—Dilute a weighed portion of the acid to a suitable volume and titrate with standard alkali solution, using phenolphthalein as indicator. About 57% of HI should be found.

1 mil of normal NaOH = 0.12793 gm. of HI, log 10697.

ACID HYDROBROMIC

HBr

Mol. Wt. 80.93

Hydrobromic acid is a clear, colorless or faintly yellowish liquid of specific gravity about 1.38. It contains at least 40% of HBr.

Under the influence of air and light the acid is likely to become discolored, due to liberation of bromine. The acid thus discolored may be decolorized by the addition of just sufficient sulphurous acid, and if the presence of the resulting sulphuric acid is objectionable, the hydrobromic acid may be freed from it by distillation.

Hydrobromic acid is used as a solvent for mercury, lead and copper and their sulphides as well as for lead sulphate; in the detection and determination of sulphur, either free or in sulphides; in detecting palm oil in margarin; and in organic synthesis.

The acid should be stored in well-closed, glass-stoppered bottles and protected from light. Owing to the readiness with which it attacks and destroys the skin it should be handled with care.

Maximum Limits of Impurities

Non-volatile Matter	0.0300%
Arsenic (As)	0.0015%
Sulphuric Acid (H_2SO_4)	0.0060%
Hydrochloric Acid (HCl)	0.1000%
Hydriodic Acid (HI)	0.0500%
Phosphorous and Phosphoric Acids (as P_2O_5)	0.0050%
Iron (Fe)	0.0006%
Other Heavy Metals	0.0000%
Calcium (Ca)	0.0050%

Methods of Testing

Non-volatile Matter.—Evaporate 10 gm. of hydrobromic acid on the steam-bath. Not more than 0.003 gm. of residue should remain.

Arsenic.—Mix 1 mil of the acid with 3 mils of stannous chloride solution and let it stand one hour. No dark color should develop.

Sulphuric Acid.—Dilute 5 mils with 50 mils of water, add barium chloride solution and let the mixture stand twelve to eighteen hours. At most 0.0005 gm. of barium sulphate should form.

Hydrochloric Acid.—Dilute 0.2 mil of the acid with 5 mils of water and add 15 mils of silver nitrate tenth-normal solution. To this mixture add 6 mils of a solution of ammonium carbonate (composed of 1 part of ammonium carbonate, 1 part of ammonia water and 3 parts of water), shake five minutes, filter and acidulate the filtrate with nitric acid. Not more than an opalescence should appear at once.

Hydriodic Acid.—To 0.25 mil of the acid add 5 mils of water, 10 mils of ammonia water and 0.1 mil of silver nitrate solution. The liquid should not acquire more than a very slight opalescence.

Phosphorous and Phosphoric Acids.—Heat to boiling 5 mils of the acid with 5 mils of nitric acid, cool the mixture, add 20 mils of ammonia water and 10 mils of magnesia mixture and allow it to stand twelve to eighteen hours. If

a precipitate forms, collect, wash, ignite and weigh it. Its weight should not exceed 0.0005 gm.

Iron.—Dilute 1 mil of the acid with 20 mils of water and add potassium sulphocyanate solution. No reddish color should develop.

Other Heavy Metals.—Dilute 5 mils of the acid with 20 mils of water and pass hydrogen sulphide gas through the mixture. No change should be apparent. Then add a slight excess of ammonia water. No dark color or precipitate should be produced.

Calcium.—Dilute 10 mils of the acid with 10 mils of water, add 15 mils of ammonia water and some ammonium oxalate solution and allow the mixture to stand one hour. No precipitate should form.

Quantitative Method.—Dilute a weighed portion of the acid to a suitable volume and titrate it with a standard alkali solution, using methyl orange as indicator. At least 40% of HBr should be found.

1 mil of normal NaOH = 0.08093 gm. of HBr, log 90806.

ACID HYDROCHLORIC, Sp. Gr. 1.18-1.19

HCl

Mol. Wt. 36.47

Hydrochloric acid, sp. gr. 1.18-1.19, is a clear, colorless, fuming liquid of pungent odor. When diluted with several volumes of water it is free from odor; odorous organic chlorides if present may be detected by such dilution. The acid has a specific gravity not below 1.18 and contains not less than 36% of HCl.

Non-volatile substances, such as alkalies, calcium, etc., are occasionally found in hydrochloric acid in quantities sufficient to require consideration in the determination of minute residues of which they constitute a part. These impurities are often derived from the glass containers in which the acid is stored, some kinds of glass being quite susceptible to corrosion.

Hydrochloric acid showing no trace of arsenic by the Marsh test upon 100 mil samples is at times available, but arsenic is difficult to remove entirely, and the acid containing minute traces of it is suitable for most reagent purposes. An acid showing a slight deposit when examined by the Marsh test may still be useful in applying tests for arsenic to other materials by less delicate methods or in the detection of more than traces of arsenic in miscellaneous substances under investigation. Several methods have been proposed for the removal of arsenic from hydrochloric acid in the laboratory. Regarding the comparative value of some of these methods see A. Coignard, Purification of Commercial Hydrochloric Acid. *Ann. Chim. anal.*, **20**, 145 (1915); *abst.*, *Chem. Abst.* **9**, 2697 (1915).

Besides the Marsh test, other methods of detecting arsenic in hydrochloric acid, reagent chemicals and other substances are of importance and deserve mention, such as the Gutzeit test and the electrolytic method. A modification of the former has been adopted in the U. S. Pharmacopoeia IX and the National Formulary for testing medicinal chemicals. Regarding the use and delicacy of these methods as well as their value as compared with The Marsh test for certain purposes see Thorpe, *J. Chem. Soc.*, **83**, 974 (1903); Report of the (British) Royal Commission on Arsenical Poisoning (1903); Kebler and Seidell, U. S. Dept. Agriculture, Bureau of Chemistry, Bulletin 105 (1906); and Scott's Standard Methods of Chemical Analysis, 2d ed., revised, 40 (1918).

Hydrochloric acid finds wide use as a general solvent, particularly for metals, their oxides, sulphides, carbonates, etc. It is a precipitant for silver, and in connection with potassium chlorate or manganese dioxide is used for generating chlorine and preparing chlorine water. It enters into many important reagents and reactions, such as Bettendorff's reagent and Gutzeit's test for arsenic, and is also employed in distinguishing between aliphatic monatomic alcohols.

Hydrochloric acid should be stored in a cool place in glass-stoppered containers as little as possible susceptible to its corrosive action. Hydrochloric acid gas is easily lost from the concentrated acid when it is exposed to even moderate summer heat. The acid is very easily discolored, especially by iron, even by minute quantities of iron, and hence great care should be exercised in protecting it from dust, cinders, organic matter, etc.

Maximum Limits of Impurities

Sulphuric Acid (H_2SO_4)	0.0002%
Non-volatile Matter	0.0005%
Free Chlorine (Cl)	0.0002%
Sulphurous Acid (SO_2)	0.0050%
Iron (Fe)	0.0001%
Other Heavy Metals	0.0000%
Arsenic (As)	0.00001%

Methods of Testing

Sulphuric Acid.—Evaporate 85 mls to a volume of about 5 mls, dilute with 20 mls of water and add barium chloride solution. No precipitate of barium sulphate should form on standing about fifteen hours.

Non-volatile Matter.—Evaporate 85 mls of the acid to dryness and ignite and weigh the residue. Not more than 0.0005 gm. should remain.

Free Chlorine.—Dilute 10 mls with 50 mls of water and add zinc iodide-starch solution. No blue color should develop within ten minutes.

Sulphurous Acid.—Add 0.05 ml of tenth-normal iodine solution and a few drops of starch paste to 50 mls of water and then add 5 mls of the hydrochloric acid, previously diluted with 50 mls of water. The mixture should retain a blue color after mixing.

Iron.—Dilute 5 mls with water to a volume of 20 mls and add potassium sulphocyanate solution. No reddish color should be produced.

Other Heavy Metals.—Pass hydrogen sulphide gas into a warm mixture of 20 mls of hydrochloric acid and 200 mls of water. No color or precipitate should be produced.

Evaporate 50 mls to about one fifth its volume, restore the loss by addition of water and add 50 mls of ammonia water and a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Arsenic.—Add 100 mls of the acid very gradually to 40 mls of nitric acid heated to about 60° C. Then add 5 mls of sulphuric acid and evaporate the mixture on a sand-bath to fumes of sulphur trioxide. Dilute the residue with 20 mls of water, introduce it into a Marsh apparatus and allow the action to proceed for one hour. At the end of this time no more than a very slight deposit of arsenic should be visible in the reduction tube.

Quantitative Method.—The specific gravity serves as a convenient index of the strength of the acid.* The acid may also be titrated with standard alkali after suitable dilution, using methyl orange as indicator. At least 36% of HCl should be found.

1 mil of normal NaOH = 0.03647 gm. of HCl, log 56194.

ACID HYDROCHLORIC, 10% (Diluted Hydrochloric Acid)

HCl

Mol. Wt. 36.47

Acid hydrochloric, 10%, is a clear, colorless liquid, having a specific gravity of about 1.05 and containing from 9.5% to 10.5% of HCl. It is a convenient acidifying agent and solvent, suitable for purposes requiring an acid weaker than that of specific gravity 1.18–1.19.

Maximum Limits of Impurities

Sulphuric Acid (H_2SO_4)	0.0001%
Non-volatile Matter	0.0005%

* For tables of specific gravity and percentage refer to page 374.

Free Chlorine (Cl)	0.0001%
Sulphurous Acid (SO ₂)	0.0020%
Iron	0.0001%
Other Heavy Metals	0.0000%
Arsenic	0.00001%

Methods of Testing

Sulphuric Acid.—Evaporate 200 mls to a volume of about 5 mls, dilute with 20 mls of water, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Non-volatile Matter.—Evaporate 100 mls of the acid to dryness and ignite and weigh the residue. The weight should not exceed 0.0005 gm.

Free Chlorine.—Dilute 25 mls of the acid with 25 mls of water, add zinc iodide-starch solution and allow to stand ten minutes. No blue color should develop.

Sulphurous Acid.—Add 0.05 ml of tenth-normal iodine solution and a few drops of starch paste to 50 mls of water and then add 15 mls of the acid, previously diluted with 35 mls of water. A blue color should persist after mixing.

Iron.—Dilute 10 mls of the acid with 10 mls of water and add potassium sulphocyanate solution. No reddish color should be produced.

Other Heavy Metals.—Pass hydrogen sulphide gas into a warm mixture of 20 mls of the acid and 100 mls of water. No change should be apparent.

Supersaturate 50 mls with ammonia water and add a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Arsenic.—To 100 mls of the acid add 10 mls of nitric acid and 5 mls of sulphuric acid, evaporate on a sand-bath to fumes of sulphur trioxide, dilute with 20 mls of water, introduce into a Marsh apparatus and allow the action to proceed for one hour. Not more than a very slight deposit of arsenic should be visible in the reduction tube.

Quantitative Method.—Determine the percentage strength of the acid in the manner described under Acid Hydrochloric, sp. gr. 1.18–1.19. From 9.5% to 10.5% of HCl should be found.

ACID HYDROFLUORIC

HF

Mol. Wt. 20.01

Hydrofluoric acid is a clear, colorless or very nearly colorless liquid. It contains at least 45% of HF.

Hydrofluoric acid fumes when exposed to the air and decreases in strength due to loss of the gaseous acid. It readily attacks and dissolves glass and other siliceous material and hence has to be stored in containers which are not susceptible to its solvent action. Hard rubber bottles sometimes used for the purpose often impart a color and an appreciable amount of soluble substances to the acid after some time.

The acid is used in the analysis of alloys and of minerals; in the separation of lead and copper from tin and antimony in electrolysis; for volatilizing vanadic acid; in the separation of niobium and tantalum; and in general for the removal or volatilization of silica.

The reagent should be kept in well-closed bottles of cersin, paraffin, vulcanite or other material not susceptible to corrosion by it. Inhalation of hydrofluoric acid fumes is dangerous and personal contact with the liquid acid should be avoided.

Maximum Limits of Impurities

Non-volatile Matter	0.0050%
Sulphuric Acid (H_2SO_4)	0.0050%
Hydrochloric Acid (HCl)	0.0050%
Hydrosilicofluoric Acid (H_2SiF_6)	0.3000%
Sulphur Dioxide (SO_2)	0.0032%
Calcium (Ca)	0.0050%
Magnesium (Mg)	0.0010%
Heavy Metals	0.0000%

Methods of Testing

In making the tests for impurities in the acid, platinum dishes should be used as far as possible.

Non-volatile Matter.—Evaporate 20 gm., ignite gently and weigh the residue. Not more than 0.001 gm. should be found.

Sulphuric Acid.—Evaporate 2 gm. on the steam-bath, take up the residue with 10 mls of water and add a few drops of nitric acid and some barium nitrate solution. No turbidity should be produced.

Hydrochloric Acid.—Dilute 2 gm. with 50 mls of water and add a few drops of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Hydrosilicofluoric Acid.—Dilute 5 gm. with 20 mls of water, add 2 mls of a cold saturated solution of potassium chloride followed by 40 mls of alcohol. No turbidity should be produced.

Sulphur Dioxide.—Dilute 10 gm. with 40 mls of water, add some starch solution and 0.05 ml of tenth-normal iodine solution. A blue color should result, persisting for more than a moment.

Calcium.—Dilute 5 gm. with 50 mls of water and add an excess of ammonia water and some ammonium oxalate solution. No turbidity should be produced at once.

Magnesium.—Dilute 5 gm. with 50 mls of water, add a slight excess of ammonia water and some ammonium phosphate solution, and allow the mixture to stand two to three hours. No precipitate should form.

Heavy Metals.—Dilute 10 gm. with 40 mls of water, warm the liquid to about 70° C. and pass into it hydrogen sulphide gas. No yellow or dark colored precipitate should form.

Dilute 5 gm. with 50 mls of water and add a slight excess of ammonia water and some ammonium sulphide solution. No precipitate or green color should be produced.

Quantitative Method.—Dilute the acid to a suitable volume and titrate with standard alkali solution, using phenolphthalein as indicator. At least 45% of HF should be found.

1 mil of normal NaOH = 0.02001 gm. of HF, log 30125.

ACID IODIC

HIO₃

Mol. Wt. 175.93

Iodic acid occurs as colorless crystals or a white crystalline powder, soluble in about 1 part of water, difficultly in alcohol and insoluble in ether. It contains at least 99.8% of HIO₃.

The reagent is used principally as an oxidizing agent; as a reagent for morphine, strychnine, emetine, naphthol, guaiacol, biliary pigments and acetoacetic acid; in organic analysis and synthesis; in the volumetric determination of mercury; and in preparing volumetric solutions.

The reagent should be stored in glass-stoppered bottles and should be protected from sunlight, by which it is decomposed.

Maximum Limits of Impurities

Non-volatile Matter	0.0250%
Insoluble Substances	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the acid and weigh the residue. The weight should not exceed 0.0005 gm.

Insoluble Substances.—Dissolve 1 gm. in 1 mil of water. The solution should be clear and colorless.

Quantitative Method.—Dry the acid over sulphuric acid to constant weight, weigh out accurately about 0.1 gm. of the dried material, dissolve it in 50 mls of water, add 2 gm. of potassium iodide and 5 mls of 10% sulphuric acid and titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 99.8% of HIO₃ should be found.

1 mil of tenth-normal Na₂S₂O₃ = 0.002932 gm. of HIO₃,
log 46716.

(ACID) IODIC ANHYDRIDE**(Iodine Pentoxide)** I_2O_5

Mol. Wt. 333.84

Iodic anhydride is usually in the form of a white, crystalline powder, soluble in water with the formation of iodic acid, HIO_3 ; insoluble in alcohol or ether. It contains at least 99.5% of I_2O_5 . On heating to about $300^\circ C$. it decomposes into iodine and oxygen. When heated in the presence of sulphur, carbon, sugar or other easily oxidizable substance, deflagration takes place.

The reagent is employed chiefly as an oxidizing agent; in the determination of carbon monoxide in the air and in gases; and for many of the purposes for which iodic acid is used. It should be stored in glass-stoppered bottles, protected from sunlight.

Maximum Limits of Impurities

Non-volatile Matter	0.0250%
Insoluble Substances	0.0000%

Methods of Testing

Non-volatile Matter and Insoluble Substances.—Perform the tests as directed under Acid Iodic. The results should be as there stated.

Quantitative Method.—Perform the quantitative determination as directed under Acid Iodic. At least 99.5% of I_2O_5 should be found.

1 mil of tenth-normal $Na_2S_2O_3 = 0.002782$ gm. of I_2O_5 ,
log 44436.

ACID LACTIC $HC_3H_5O_3$

Mol. Wt. 90.05

Lactic acid is a clear, colorless or slightly yellowish, odorless or nearly odorless, syrupy liquid. It is miscible with water, alcohol or ether but not with chloroform, benzin or carbon disulphide. The acid is hygroscopic and optically

inactive. It contains at least 85% of $\text{HC}_2\text{H}_3\text{O}_2$ and has a specific gravity of about 1.21.

Lactic acid ordinarily contains varying amounts, sometimes as high as 8 to 10%, of lactic anhydride, formed during concentration of the acid. This is not susceptible to titration until hydrolyzed. The quantitative method under the methods of testing provides for the hydrolysis of the anhydride and its subsequent titration as lactic acid. The percentage requirement of 85% includes the acid derived from the anhydride.

Lactic acid is used principally as a constituent of alkaline copper solutions for the detection and determination of reducing sugars; for the detection of pyrogallol and oil of savin; in differentiating phenol and resorcin from salicylic acid; and in the electrolytic determination of cobalt and nickel.

The acid should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Non-volatile Matter	0.1000%
Hydrochloric Acid (HCl)	0.0100%
Sulphuric Acid (H_2SO_4)	0.0100%
Iron (Fe)	0.0010%
Other Heavy Metals	0.0000%
Substances Reducing Alkaline Copper Solution	0.0000%
Organic Impurities	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 5 mls of the acid and weigh the ash. Not more than 0.006 gm. should be found.

Hydrochloric Acid.—To 10 mls of the 1% solution of the acid add a few drops of silver nitrate solution. No change should be apparent.

Sulphuric Acid.—Add about 1 mil of barium chloride solution to 10 mls of a 5% solution of lactic acid. No change should be apparent.

Iron and Other Heavy Metals.—Dilute 1 mil with 20 mls of water and add hydrogen sulphide water. No change should be apparent.

Dilute 1 mil with 20 mils of water and add a slight excess of ammonia water and a few drops of ammonium sulphide solution. A slight green color may develop but no precipitate or dark brown color should be produced.

Substances Reducing Alkaline Copper Solution.—To 5 mils of the lactic acid add a slight excess of sodium hydroxide solution and then add 10 mils of Fehling's alkaline copper sulphate solution. Boil the liquid for two or three minutes and allow it to stand one to two hours. No precipitate of cuprous oxide should form.

Organic Impurities.—Carefully overlay 5 mils of sulphuric acid with 5 mils of lactic acid, keeping the temperature of the liquids at or a little below 15° C., and allow to stand at that temperature for ten minutes. No dark color should develop at the plane of contact of the liquids.

Quantitative Method.—Weigh accurately about 2.5 mils of the acid, add 50 mils of normal sodium hydroxide solution, boil for twenty minutes, replacing the water lost by evaporation, and then titrate the boiling solution with normal solution of sulphuric acid, using phenolphthalein as indicator. At least 85% of $\text{HC}_3\text{H}_5\text{O}_3$ should be found.

1 mil of normal NaOH = 0.09005 gm. of $\text{HC}_3\text{H}_5\text{O}_3$,
log 95448.

ACID MOLYBDIC, 85%

Molybdic acid, 85%, is usually a white or slightly yellowish powder, soluble in ammonia water and in solutions of the fixed alkalis. It contains at least 83% and usually about 85% of MoO_3 .

Although the formula H_2MoO_4 is sometimes assigned to it, its composition is somewhat variable, considerable amounts of ammonium salts often being present as well as nitrates and at times also alkalis and sulphates. These, however, do not interfere with its ordinary uses. When molybdic acid is required free from these substances, (Acid) Molybdic Anhydride should be used.

Molybdic acid is used largely in the determination of phosphates and also in determining arsenates, lead and bismuth; as a reagent for hydrogen peroxide, albumin, alcohols and phenols; and for detecting water in alcohol and ether.

The reagent should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Heavy Metals	0.0000%
Phosphates (P_2O_5)	0.0005%

Methods of Testing

Insoluble Substances.—Dissolve 2 gm. of molybdic acid in 15 mls of ammonia water. No insoluble residue should be left.

Heavy Metals.—To the solution obtained in the above test add hydrogen sulphide water. No dark brown color or precipitate should be produced although the solution may become greenish-yellow.

Phosphates.—Dissolve 10 gm. of molybdic acid in 25 mls of ammonia water, pour this solution gradually with stirring into 150 mls of nitric acid and allow the mixture to stand twelve to eighteen hours at about 40° C. No yellow precipitate should form.

Quantitative Method.—Dissolve about 0.5 gm. accurately weighed, in a mixture of 50 mls of water and 3 mls of ammonia water with the aid of a gentle heat. Acidulate the solution with 5 mls of 36% acetic acid, dilute to 200 mls with water, heat to boiling and add a clear solution of 1.5 gm. of lead acetate, crystals, in 20 mls of water. Boil for several minutes with constant stirring until the precipitate becomes granular and settles readily to the bottom of the vessel. After waiting a minute or two for the precipitate to settle, decant the supernatant liquid through a tared filter (a Gooch filter with a fairly thick pad of asbestos, previously ignited, is best), wash the precipitate

by decantation ten times with 50-mil portions of boiling water, finally remove the precipitate to the filter, and after the water has drained off, ignite and weigh it as PbMoO_4 . At least 83% of MoO_3 should be found.

$$\text{PbMoO}_4 \times 0.39215 = \text{MoO}_3, \text{ log } 59345$$

(ACID) MOLYBDIC ANHYDRIDE

(Molybdic Acid, 100%)

MoO_3 ,

Mol. Wt. 144.00

Molybdic anhydride is a powder, usually slightly yellowish but at times slightly bluish in color. It is soluble in ammonia water and in solutions of the fixed alkalis. It contains at least 99.5% of MoO_3 .

The bluish color sometimes observed is due to the presence of some of the lower oxides of molybdenum, which also tend to raise the percentage of molybdenum present, thus accounting for assays of the acid exceeding 100% when calculated as MoO_3 .

It may be used for the same purposes as Acid Molybdic, 85%, as well as for others requiring absence of ammonia or nitrates. It is a constituent of Froehde's reagent for testing alkaloids.

The reagent should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Alkalis	0.0000%
Ammonium Salts (NH_4)	0.0035%
Heavy Metals	0.0000%
Phosphates (P_2O_5)	0.0005%
Nitrates (N_2O_5)	0.0032%
Sulphates (SO_4)	0.0500%
Chlorides (Cl)	0.0020%
Insoluble Matter	0.0000%

Methods of Testing

Alkalis.—Heat 1 gm. in a test-tube. The molybdic anhydride should not fuse to a dark-colored mass below a

red heat. At that temperature it should only begin to melt at the point of contact with the tube wall, while a crystalline sublimate should form upon the cooler surface of the tube.

Ammonium Salts.—Boil 1 gm. with 10 mls of 27% sodium hydroxide solution. The vapors should not turn moistened red litmus paper blue.

Heavy Metals.—Dissolve 5 gm. in 40 mls of ammonia water, add hydrogen sulphide water and allow it to stand ten minutes. No precipitate should form nor should the liquid be darker than pale yellow.

Phosphates.—Dissolve 10 gm. in 25 mls of ammonia water, pour this solution gradually and with stirring into 150 mls of nitric acid, and let the mixture stand for twelve to eighteen hours at about 40° C. No yellow precipitate should form.

Nitrates.—Shake 1 gm. with 10 mls of water, add a few milligrams of sodium chloride and 1 drop of indigo solution and mix with 10 mls of sulphuric acid. The blue color imparted by the indigo should persist for a few moments at least after mixing.

Sulphates and Chlorides.—Dissolve 10 gm. in 30 mls of ammonia water and pour the solution slowly and with stirring into 50 mls of nitric acid. Add to the clear mixture silver nitrate and barium nitrate solutions. No turbidity should be produced, and after standing twelve to eighteen hours no precipitate of barium sulphate should form.

Insoluble Matter.—Dissolve 5 gm. of molybdic anhydride in 40 mls of ammonia water, heating gently if necessary. No insoluble residue should remain.

Quantitative Method.—The determination of MoO_3 is carried out as described under Acid Molybdic, 85%. At least 99.5% of MoO_3 should be found.

ACID NAPHTHYLAMINESULPHONIC

(Alphanaphthylaminesulphonic Acid; Naphthionic Acid)



Mol. Wt. 464.40

Naphthylaminesulphonic acid is a white powder, or small, lustrous, colorless needles (when crystallized from hot water), which carbonize but do not melt on being heated. It is soluble in about 4000 parts of cold water, more readily in hot water, scarcely soluble in alcohol, and almost insoluble in ether; alkaline fluids dissolve it easily. The solution in ammonia water exhibits a violet fluorescence.

The reagent is used for the detection and colorimetric determination of small quantities of nitrous acid. It may be employed by adding 20 to 30 mgm. to 5 mls of the solution to be tested, followed by 2 or 3 drops of hydrochloric acid. After thorough shaking 1 or 2 mls of ammonia water are added, the presence of nitrites being indicated by the production of a red or reddish color.

Naphthylaminesulphonic acid should be stored in well-stoppered, amber-colored bottles.

ACID NITRIC, Sp. Gr. 1.40-1.42

Mol. Wt. 63.02

Nitric acid, sp. gr. 1.40-1.42, is a clear, colorless, or at most faintly yellowish liquid of specific gravity about 1.40 to 1.42, and containing at least 65% and usually not more than 70% of HNO_3 .

The yellowish color frequently observed in the acid is due to the presence of hyponitric acid, N_2O_4 , which is nearly always present to some extent, especially in concentrated nitric acid, and usually results from exposure of the acid to sunlight or heat. Nitric acid containing a large quantity of hyponitric acid is known as fuming nitric acid.

Nitric acid has wide use as a solvent, especially for metals, as an oxidizing agent and in the nitration of organic compounds. In connection with potassium chlorate it is

used in the determination of sulphur and phosphorus in organic bodies, and alone in the Carius method of determining sulphur, phosphorus and halogens.

On account of its strong oxidizing power the possibility of accidental contact with organic matter, such as paper, excelsior and similar packing material should be carefully avoided, as well as contact with the person in handling the acid. It is best stored in glass-stoppered containers in a cool place and should be protected from light, especially direct sunlight. Bottles containing the acid should be of amber-colored glass.

Maximum Limits of Impurities

Non-volatile Matter	0.0035%
Sulphuric Acid (H_2SO_4)	0.0006%
Hydrochloric Acid (HCl)	0.0001%
Iodic Acid and Iodine (I)	0.0005%
Heavy Metals	0.0000%
Calcium (Ca)	0.0020%
Arsenic (As)	0.00001%

Methods of Testing

Non-volatile Matter.—Evaporate 20 mls of the nitric acid and ignite and weigh the residue. Not more than 0.001 gm. should remain.

Owing to its highly corrosive nature, nitric acid easily acquires non-volatile substances by solution, which appear in the determination of residue.

Sulphuric Acid.—Evaporate to dryness 25 mls of the acid to which about 0.1 gm. of sodium carbonate has been added, dissolve the residue in 50 mls of water, add barium chloride solution and let it stand fifteen hours. Any precipitate forming in this time should not weigh more than 0.0005 gm.

Hydrochloric Acid.—Dilute 20 mls of the acid with 50 mls of water and add silver nitrate solution. No change should be apparent.

Iodic Acid and Iodine.—Dilute 20 mls of the acid with 20 mls of water, add a small piece of zinc and a few mls of

chloroform and shake the mixture. The chloroform should not acquire a violet color.

Heavy Metals.—Dilute 25 mls of the acid with 50 mls of water and add ammonia water in slight excess and a few drops of ammonium sulphide solution. No precipitate should be produced nor should the liquid acquire a green or brown color.

Calcium.—Dilute 10 mls of the acid with 50 mls of water and add ammonia water in slight excess followed by ammonium oxalate solution. No turbidity should be produced.

Arsenic.—Mix 50 mls of nitric acid with 5 mls of sulphuric acid, evaporate the mixture on a sand bath to fumes of sulphur trioxide, dissolve the residue in 20 mls of water and introduce it into a Marsh apparatus. After the action has proceeded one hour no deposit of arsenic should be visible in the reduction tube.

Quantitative Method.—The specific gravity furnishes a convenient means of determining the strength of the acid.* It may also be titrated, after suitable dilution, with standard alkali solution, using methyl orange as indicator. At least 65% of HNO_3 should be found.

1 mil of normal NaOH = 0.06302 gm. of HNO_3 , log 79948.

ACID NITRIC, Sp. Gr. 1.20

HNO_3

Mol. Wt. 63.02

Nitric acid, sp. gr. 1.20, is a clear, colorless liquid of specific gravity about 1.20 and containing at least 32% and usually not more than 34% of HNO_3 .

It is used as a solvent and acidifying reagent. It is not so liable to decomposition by the action of heat and light as the acid of specific gravity 1.40–1.42 although similar precautions as to storage are advisable for both.

* For tables of specific gravity and percentage of nitric acid refer to page 375.

Maximum Limits of Impurities

Non-volatile Matter	0.0020%
Sulphuric Acid (H_2SO_4)	0.0003%
Hydrochloric Acid (HCl)	0.0001%
Iodic Acid and Iodine (I)	0.0003%
Heavy Metals	0.0000%
Calcium (Ca)	0.0010%
Arsenic (As)	0.00001%

Methods of Testing

Non-volatile Matter.—Evaporate 20 mls of the acid and ignite and weigh the residue. Not more than 0.0005 gm. should be found.

Sulphuric Acid.—Evaporate to dryness 60 mls of the acid to which about 0.1 gm. of sodium carbonate has been added, dissolve the residue in 50 mls of water, add barium chloride solution and let it stand at least twelve hours. Any precipitate forming should not weigh over 0.0005 gm.

Hydrochloric Acid.—To 25 mls add silver nitrate solution. No change should be apparent.

Iodic Acid and Iodine.—To 40 mls of the acid add a small piece of zinc and a few mls of chloroform. On shaking, the chloroform should not acquire a violet color.

Heavy Metals.—Add ammonia water in slight excess to 50 mls of the acid and add a few drops of ammonium sulphide solution. No precipitate should be produced, nor should the liquid become green or brown.

Calcium.—Add ammonia water in slight excess to 20 mls of the acid, dilute it to about 100 mls and add ammonium oxalate solution. No turbidity should be produced.

Arsenic.—Mix 75 mls of the acid with 5 mls of sulphuric acid, evaporate the mixture on a sand bath to fumes of sulphur trioxide, dissolve the residue in 20 mls of water and introduce it into a Marsh apparatus. After the action has proceeded for one hour no arsenic mirror should be visible in the reduction tube.

Quantitative Method.—Titrate the acid, after suitable

dilution, with standard alkali solution, using methyl orange as indicator. At least 32% of HNO_3 should be found.

1 mil of normal NaOH = 0.06302 gm. of HNO_3 , log 79948.

The strength of the acid may also be ascertained from the specific gravity by reference to the table on page 375.

ACID NITRIC, 10%

HNO_3

Mol. Wt. 63.02

Nitric acid, 10%, is a clear, colorless liquid of specific gravity about 1.06 and contains from 9.5% to 10.5% of HNO_3 . It is conveniently used as an acidifying agent and solvent when a stronger acid is unnecessary. It is the official test solution of the U. S. Pharmacopœia, 9th Revision.

Maximum Limits of Impurities

Non-volatile Matter	0.0005%
Sulphuric Acid (H_2SO_4)	0.0002%
Hydrochloric Acid (HCl)	0.0001%
Iodic Acid and Iodine (I)	0.0004%
Heavy Metals	0.0000%
Calcium (Ca)	0.0003%
Arsenic (As)	0.00001%

Methods of Testing

Non-volatile Matter.—Evaporate 95 mls of the acid and ignite and weigh the residue. Not more than 0.0005 gm. should be found.

Sulphuric Acid.—Evaporate to dryness 100 mls of the acid to which about 0.1 gm. of sodium carbonate has been added, dissolve the residue in 50 mls of water, add barium chloride solution and let it stand twelve to eighteen hours. Any precipitate forming should not weigh over 0.0005 gm.

Hydrochloric Acid.—Add to 100 mls of the acid some silver nitrate solution. No change should be apparent.

Iodic Acid and Iodine.—To 35 mls of the acid add a small piece of zinc and a few mls of chloroform. On shaking, the chloroform should not acquire a violet color.

Heavy Metals.—Add ammonia water in slight excess to 100 mls of the acid and then add a few drops of ammonium sulphide solution. No precipitate should form and no green or brown color should be produced.

Calcium.—Add a slight excess of ammonia water to 100 mls of the acid and then add ammonium oxalate solution. No turbidity should be produced.

Arsenic.—Mix 100 mls of the acid with 5 mls of sulphuric acid, evaporate the mixture on a sand-bath to fumes of sulphur trioxide, dissolve the residue in 20 mls of water and introduce it into a Marsh apparatus. After the action has proceeded for one hour no deposit of arsenic should be visible.

Quantitative Method.—Titrate the acid with standard alkali solution, using methyl orange as indicator. At least 9.5% and not more than 10.5% of HNO_3 should be found.

1 mil of normal NaOH = 0.06302 gm. of HNO_3 , log 79948.

ACID NITRIC, FUMING

Fuming nitric acid for reagent use is a clear, yellowish to brownish-red, fuming liquid of specific gravity about 1.50. It consists of nitric acid, HNO_3 , containing more or less hyponitric acid, N_2O_4 , and other oxides of nitrogen.

The color of the fuming nitric acid varies with the quantity of dissolved nitrogen oxides, larger amounts producing darker colors. Nitric acid is said to be capable of dissolving as much as 35% of N_2O_4 although the fuming acid of the market contains usually much less than this. The composition of the acid can not be ascertained directly from its specific gravity but the percentage of N_2O_4 present may be determined by titration with permanganate and, by referring to tables, the ascertained specific gravity may be corrected for the amount of N_2O_4 found. From the corrected specific gravity the percentage of HNO_3 may then be obtained by reference to the customary nitric acid specific gravity tables. For details of the method of titrating the N_2O_4 and tables for correcting the specific gravity see

Lunge's Technical Methods of Chemical Analysis, translation by C. A. Keane, 1, 324, 328 (1908).

Fuming nitric acid is a more energetic oxidizing agent than the acid of specific gravity 1.40–1.42 and is therefore often employed for purposes requiring the destruction of refractory organic material, as in the determination of sulphur, phosphorus and halogens by the Carius method.

Fuming nitric acid should be stored in glass-stoppered bottles in a cool place. For fire protection contact with organic material, such as straw, paper, sawdust, etc., must be avoided, and for personal safety great care should be exercised in handling the acid. Inhalation of the fumes is dangerous.

Maximum Limits of Impurities

Non-volatile Matter	0.0033%
Sulphuric Acid (H_2SO_4)	0.0010%
Hydrochloric Acid (HCl)	0.0002%
Iodic Acid and Iodine (I)	0.0008%
Heavy Metals	0.0000%
Calcium (Ca)	0.0004%
Arsenic (As)	0.00002%

Methods of Testing

Non-volatile Matter.—Evaporate 20 mls of fuming nitric acid and ignite the residue. Not more than 0.001 gm. should remain.

Sulphuric Acid.—Evaporate to dryness 15 mls of the acid to which about 0.1 gm. of sodium carbonate has been added, dissolve the residue in 50 mls of water, add barium chloride solution and let it stand about fifteen hours. Any precipitate forming in this time should not weigh more than 0.0005 gm.

Hydrochloric Acid.—Dilute 30 mls of the acid to 100 mls and add silver nitrate solution. Not more than a slight opalescence should be produced.

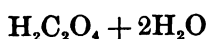
Iodic Acid and Iodine.—Dilute 12 mls of the acid with 25 mls of water, add a small piece of zinc and a few mls of chloroform. On shaking, the chloroform should not acquire a violet color.

Heavy Metals.—Dilute 15 mls of the acid with 60 mls of water, add ammonia water in slight excess followed by a few drops of ammonium sulphide solution. No precipitate should be produced nor should the liquid acquire a green or brown color.

Calcium.—Dilute 10 mls of the acid with 60 mls of water, add ammonia water in slight excess and then add ammonium oxalate solution. No turbidity should be produced.

Arsenic.—Mix 25 mls of the acid with 5 mls of sulphuric acid, evaporate the mixture on a sand-bath to fumes of sulphur trioxide, dissolve the residue in 20 mls of water and introduce it into a Marsh apparatus. After the action has proceeded one hour no arsenic mirror should be visible in the reduction tube.

ACID OXALIC



Mol. Wt. 126.06

Oxalic acid usually occurs as colorless crystals, efflorescent in dry air. It is soluble in about 10 parts of water and in about 2.5 parts of alcohol. The reagent contains not less than 99.8% of $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$.

When heated the hydrated acid first becomes anhydrous and then at about 100° C. sublimes. The sublimed acid has a melting point of about 187° C. and has been employed as a starting material for volumetric solutions, but is not well adapted to this purpose as it is very hygroscopic.

Oxalic acid is used chiefly for the preparation of volumetric solutions for alkalimetry and oxidimetry; as a reducing agent, as in the precipitation of gold; in the separation of alkalis, magnesium and the rarer earths (cerium, zirconium, thorium, etc.); in the detection and determination of calcium, lead peroxide and manganese dioxide; in the electro-analysis of copper, tin and cadmium; and as a reagent for indol, hydrogen peroxide, colchicine, morphine and urea.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Ash	0.0200%
Sulphates (SO_4)	0.0040%
Chlorides (Cl)	0.0020%
Nitrates (N_2O_5)	0.0070%
Heavy Metals	0.0000%
Ammonium Compounds (NH_3)	0.0020%

Methods of Testing

Ash.—Ignite 5 gm. of oxalic acid and weigh the residue. The weight should not exceed 0.001 gm.

Sulphates.—Dissolve 5 gm. in 100 mls of water, add 1 mil each of hydrochloric acid and of barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 5 gm. in 75 mls of water and add 15 mls of nitric acid and a few drops of silver nitrate solution. Not more than slight opalescence should be produced.

Nitrates.—Dissolve 1 gm. in 10 mls of water and overlay the solution upon 10 mls of diphenylamine solution. No blue color should develop at the plane of contact of the liquids.

Heavy Metals.—Dissolve 3 gm. in 30 mls of water and add hydrogen sulphide water. No change should be apparent. Then add ammonia water in slight excess and a few drops of ammonium sulphide solution. No green or brown color should be produced nor should a precipitate form.

Ammonium Compounds.—Dissolve 2.5 gm. of the oxalic acid and 5 gm. of potassium hydroxide in 30 mls of water and add 1 mil of Nessler's solution. No brownish-red color should develop although the liquid may become slightly yellow.

Quantitative Method.—Dissolve the acid in water and titrate the solution with standard alkali solution using phenolphthalein as indicator. Dissolve another portion of the acid in water, add a few mls of sulphuric acid, heat to about

60° C. and titrate with tenth-normal potassium permanganate solution. At least 99.8% of $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$ should be found in each titration.

1 mil of normal $\text{NaOH} = 0.06303$ gm. of $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$,
log 79955.

1 mil of tenth-normal $\text{KMnO}_4 = 0.006303$ gm. of
 $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$, log 79955.

ACID PERCHLORIC

HClO_4

Mol. Wt. 100.47

Perchloric acid is a colorless liquid. It has a specific gravity of about 1.15 and contains not less than 20% of HClO_4 .

The acid finds its principal uses in the detection and determination of potassium; in electro-analysis, especially in the deposition of silver, copper and cadmium; as an alkaloidal reagent; and in the destruction of organic matter in forensic and other analyses.

Perchloric acid should be kept in glass-stoppered bottles.

Maximum Limits of Impurities

Non-volatile Matter	0.0200%
Sulphuric Acid (H_2SO_4)	0.0060%
Hydrochloric Acid (HCl)	0.0010%
Barium (Ba)	0.0025%
Heavy Metals	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 10 gm. of the acid and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Sulphuric Acid.—Dilute 5 mils with 100 mils of water, add 5 mils of hydrochloric acid and some barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Hydrochloric Acid.—Dilute 5 mils with 25 mils of water and add 2 or 3 mils of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Barium.—Dilute 10 mls of the acid with 50 mls of water, add a little diluted sulphuric acid and allow to stand five minutes. No turbidity should result.

Heavy Metals.—Dilute 1 ml with water to 10 mls and add hydrogen sulphide water. No change should be apparent. Make the solution slightly ammonical and add a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Quantitative Method.—Titrate the acid, after suitable dilution, with standard alkali solution, using phenolphthalein as indicator. Not less than 20% of HClO_4 should be found.

1 ml of normal $\text{NaOH} = 0.10047$ gm. of HClO_4 , log 00204.

ACID PHOSPHOMOLYBDIC

$20\text{MoO}_3 \cdot 2\text{H}_3\text{PO}_4 + 48\text{H}_2\text{O}$

Mol. Wt. 3940.90

Phosphomolybdic acid usually occurs as lustrous, yellow crystals, easily soluble in water. Its solutions are acid to litmus. From strongly acid solutions phosphomolybdic acid precipitates potassium, ammonium, rubidium, caesium, tantalum and alkaloids, but not sodium or lithium. Alkalis decompose the acid forming molybdates and phosphomolybdates of higher phosphorus content.

The reagent is used for the detection of the bases mentioned above which form insoluble compounds with it; as a reagent for uric acid, xanthin, creatinine, digitalin and morphine; and in the detection and determination of vanadium.

Phosphomolybdic acid should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Heavy Metals	Trace
Calcium (Ca)	0.0200%

Methods of Testing

Insoluble Matter.—Gently warm 1 gm. of phosphomolybdic acid with 10 mls of water. The acid should dissolve completely.

Heavy Metals.—To the solution obtained in the preceding test add ammonia water until the precipitate first formed is redissolved and then add a few drops of ammonium sulphide solution. Not more than a slight green color should be produced.

Calcium.—Dissolve 1 gm. in 10 mls of water with the aid of heat, add an excess of ammonia water and some ammonium oxalate solution. No change should be apparent.

ACID PHOSPHORIC, 85%

(Orthophosphoric Acid)

H_3PO_4

Mol. Wt. 98.06

Acid phosphoric, 85%, is a clear, syrupy liquid, colorless or very nearly so. It is clearly miscible with water and with alcohol. It has a specific gravity not lower than 1.710 and contains not less than 85% of H_3PO_4 . The acid readily takes up moisture when exposed to the air.

Phosphoric acid of high concentration frequently becomes slightly colored from traces of iron which it is prone to retain or from slight contact with organic or other foreign matter. The acid, however, should appear colorless when viewed in a layer of 15 to 20 mm. held against a white background. A slight color does not interfere with its ordinary uses.

Phosphoric acid is used principally in the separation of volatile acids from their salts by distillation; also as a reagent for alkaloids, bile-acids, and albumin.

The acid should be stored in well-closed, glass-stoppered bottles in a moderately warm place. Crystals of phosphoric acid often separate from the syrupy acid when exposed to cold, leaving a supernatant acid of somewhat lower strength. The crystals slowly redissolve on warming the acid.

Maximum Limits of Impurities

Arsenic (As)	0.0002%
Volatile Acids (as $\text{HC}_2\text{H}_3\text{O}_2$)	0.0014%
Nitric Acid (HNO_3)	0.0020%
Hydrogen Halogen Acids (as HCl)	0.0003%
Sulphuric Acid (H_2SO_4)	0.0030%
Metaphosphoric Acid (HPO_3)	0.0000%
Heavy Metals	Traces
Calcium (Ca)	0.0060%
Substances Insoluble in Alcohol	0.0000%
Substances Reducing Permanganate (as H_3PO_3) ...	0.0120%

Methods of Testing

Arsenic.—Mix 3 mls of the acid with 20 mls of water, introduce it a little at a time into a Marsh apparatus and allow the action to proceed for one hour.. No deposit of arsenic should be visible in the reduction tube.

Volatile Acids.—Dilute 25 mls of the acid with 50 mls of water, distil the liquid, collect 50 mls of distillate and titrate it with tenth-normal sodium hydroxide solution, using methyl orange as indicator. Not more than 0.1 mil should be required to obtain the end-point.

Nitric Acid.—Dilute 1 mil with 10 mls of water, add 1 drop of indigo solution and a few milligrams of sodium chloride and mix the liquid with 10 mls of sulphuric acid. The blue color should persist after mixing.

Hydrogen-Halogen Acids.—Mix 2 mls of the acid with 18 mls of water and add silver nitrate solution. No change should be apparent.

Sulphuric Acid.—Dilute 2 mls of the acid to a volume of 20 mls, add barium chloride solution and allow it to stand two hours. No precipitate of barium sulphate should form.

Metaphosphoric Acid.—Dilute the acid with 10 volumes of water and add it by drops to a dilute solution of albumen. No turbidity should be produced.

Heavy Metals.—Dilute 2 mls of the acid to a volume of 10 mls and add 10 mls of hydrogen sulphide water. No

change should be apparent. Add further a slight excess of ammonia water and a few drops of ammonium sulphide solution. No precipitate should be produced, although a slight greenish color may develop.

Calcium.—Dilute 2 mls of the acid to a volume of 20 mls and add a slight excess of ammonia water and some ammonium oxalate solution. No precipitate should form.

Substances Insoluble in Alcohol.—Mix 5 mls of the acid with 20 mls of absolute alcohol. The mixture should be clear.

Substances Reducing Permanganate (Phosphorous Acid, etc.).—Mix 5 mls of the acid with 5 mls of diluted sulphuric acid, add 0.25 ml of tenth-normal potassium permanganate solution and heat the mixture in a steam-bath at about 100° C. for five minutes. The red color of the liquid should not be entirely discharged.

Quantitative Method.—A specific gravity not below 1.710 insures a content of at least 85% of H_3PO_4 .

The acid content may also be determined volumetrically as follows: Dilute a weighed portion of the acid to a convenient volume and take for analysis an aliquot representing about 0.1 gm. of the original acid. Place it in a 100-ml graduated flask, add a drop of phenolphthalein solution and neutralize it with 5% sodium hydroxide solution (free from chloride). Then introduce 50 mls of tenth-normal silver nitrate solution, agitate the liquid and gradually add zinc oxide (free from chloride) until the liquid is neutral to litmus. Dilute the mixture to 100 mls and, after mixing thoroughly, filter. To 50 mls of the filtrate add 2 or 3 mls each of nitric acid and 10% solution of ferric ammonium sulphate and titrate with tenth-normal ammonium sulphocyanate solution. At least 85% of H_3PO_4 should be found.

1 ml of tenth-normal $AgNO_3 = 0.0032687$ gm. of H_3PO_4 ,
log 51437.

ACID PHOSPHORIC, 10%**H₃PO₄**

Mol. Wt. 98.06

Acid phosphoric, 10%, is a clear, colorless, odorless liquid, of specific gravity about 1.057 and contains not less than 9.5% and not more than 10.5% of H₃PO₄. It is clearly miscible with water and with alcohol. The acid should be stored in glass-stoppered bottles.

Maximum Limits of Impurities

Arsenic (As)	0.00005%
Volatile Acids (as HC ₂ H ₃ O ₂)	0.0005%
Nitric Acid (HNO ₃)	0.0003%
Hydrogen-Halogen Acids (as HCl)	0.0001%
Sulphuric Acid (H ₂ SO ₄)	0.0004%
Metaphosphoric Acid (HPO ₃)	0.0000%
Heavy Metals	Traces
Calcium (Ca)	0.0010%
Substances Insoluble in Alcohol	0.0000%
Substances Reducing Permanganate (as H ₂ PO ₃) ...	0.0020%

Methods of Testing

Arsenic.—Introduce 20 mls, a little at a time, into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

Volatile Acids.—Distil 60 mls of the acid, collect 50 mls of distillate and titrate it with tenth-normal sodium hydroxide solution, using methyl orange as indicator. Not more than 0.05 ml should be required.

Nitric Acid.—To 10 mls of the acid add 1 drop of indigo solution and a few milligrams of sodium chloride and mix the liquid with 10 mls of sulphuric acid. The blue color should persist after mixing.

Hydrogen-Halogen Acids.—To 20 mls of the acid add silver nitrate solution. No change should be apparent.

Sulphuric Acid.—To 25 mls of the acid add barium chloride solution and allow it to stand two hours. No precipitate of barium sulphate should form.

Metaphosphoric Acid.—Add the acid by drops to a dilute solution of albumin. No turbidity should be produced.

Heavy Metals.—To 10 mls of the acid add 10 mls of hydrogen sulphide water. No change should be apparent. Add further a slight excess of ammonia water and a few drops of ammonium sulphide solution. No precipitate should form, although a slight greenish color may develop.

Calcium.—To 20 mls of the acid add a slight excess of ammonia water and some ammonium oxalate solution. No precipitate should form.

Substances Insoluble in Alcohol.—To 10 mls of the acid add 50 mls of absolute alcohol. The mixture should be clear.

Substance Reducing Permanganate.—Heat 50 mls of the acid to boiling, add 0.25 mls of tenth-normal potassium permanganate solution and allow it to stand for five minutes on a steam-bath. The red color of the liquid should not be entirely discharged.

Quantitative Method.—The strength of the acid may be ascertained most conveniently by taking the specific gravity and referring to phosphoric acid specific gravity tables.

The phosphoric acid may also be determined by the volumetric method described under Acid Phosphoric, 85%: At least 9.5% and not more than 10.5% of H_3PO_4 should be found.

(ACID) PHOSPHORIC ANHYDRIDE

(Phosphorus Pentoxide)

P_2O_5

Mol. Wt. 142.08

Phosphoric anhydride is a white, very deliquescent, amorphous, bulky powder, soluble in water with a hissing noise and the generation of heat, forming metaphosphoric acid. On heating it sublimes. Phosphoric anhydride frequently contains lower oxides of phosphorus, detected by

their reducing action upon silver nitrate or mercuric chloride solution.

The employment of phosphorus pentoxide in the laboratory is based upon its great affinity for water, it being able to abstract even the elements of water from organic bodies. It is used mainly in organic synthesis as a dehydrating agent and is also useful for drying gases and in nitrogen determinations by the Kjeldahl method.

Phosphoric anhydride should be kept in well-closed, glass-stoppered bottles.

Maximum Limit of Impurity

Arsenous Acid (As_2O_3) 0.0100%

Method of Testing

Arsenous Acid.—Introduce 1 gm., in small portions at a time, into 20 mls of water, warm the solution and pass into it a current of hydrogen sulphide gas. The liquid may acquire a slightly yellow color but no yellow precipitate should form.

ACID PHOSPHORIC, META

(Glacial Phosphoric Acid)

HPO_3 ,

Mol. Wt. 80.05

Acid phosphoric, meta, usually occurs as colorless, glassy, cylindrical sticks or pieces, easily soluble in water. It is deliquescent in moist air. Pure metaphosphoric acid is a soft, gelatinous substance, becoming liquid when heated. The hard, vitreous appearance of the reagent is due to the presence of sodium metaphosphate, added as a preservative and to facilitate handling. The presence of this salt does not interfere with the ordinary uses of the reagent.

Aqueous solutions of metaphosphoric acid are not stable, the acid soon changing, especially with heat, to orthophosphoric acid. This change is hastened by the presence of strong mineral acids and retarded by weak acids (*e. g.*, acetic acid) or by neutralization.

Metaphosphoric acid is used chiefly for detecting albumin; detecting soap in lubricating oils; and in preparing dental cements. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Nitric Acid (HNO_3)	0.0200%
Sulphuric Acid (H_2SO_4)	0.0120%
Hydrochloric Acid (HCl)	0.0050%
Iron (Fe)	0.0150%
Other Heavy Metals	0.0000%
Calcium (Ca)	0.0200%
Arsenic (As)	0.0015%
Oxidizable Substances (as H_2PO_3)	0.0410%

Methods of Testing

Nitric Acid.—Dissolve 1 gm. in 2 mls of water, add 2 mls of sulphuric acid and overlay the mixture with 1 mil of ferrous sulphate solution. No brown color should develop at the plane of contact of the liquids.

Sulphuric Acid.—Dissolve 1 gm. in 20 mls of water and add 5 mls of hydrochloric acid and some barium chloride solution. There should be no turbidity produced at once.

Hydrochloric Acid.—Dissolve 1 gm. in 20 mls of water and add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Iron.—Dissolve 1 gm. in 20 mls of water, add an excess of ammonia water and a few drops of ammonium sulphide solution. A greenish color may appear but no precipitate should be produced at once.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No color or precipitate should be produced.

Calcium.—Dissolve 1 gm. in 20 mls of water and add an excess of ammonia water and some ammonium oxalate solution. No precipitate should result.

Arsenic.—Dissolve 1 gm. in 1 mil of water, add 5 mls of stannous chloride solution and allow the mixture to stand one hour. No dark color should develop.

Oxidizable Substances.—Dissolve 1 gm. in 10 mils of water, add 5 mils of 10% sulphuric acid and 0.1 mil of tenth-normal potassium permanganate solution and heat for five minutes on a steam-bath. The pink color of the mixture should not disappear.

ACID PHOSPHOTUNGSTIC



Phosphotungstic acid usually occurs as small, white or slightly yellowish-green crystals or crystalline powder, easily soluble in water, forming solutions acid to litmus. It is slightly efflorescent. The water content of the reagent is somewhat variable and uncertain.

The acid precipitates from strongly acid solutions insoluble phosphotungstates of potassium, ammonium and of many alkaloids and nitrogenous organic bases. It is used chiefly as a reagent for potassium, ammonium, alkaloids, uric acid, albumins, albumoses, peptones, blood and spermatic fluid.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Nitric Acid (HNO_3)	0.0038%
Ammonium Salts (NH_4)	0.0045%

Methods of Testing

Nitric Acid.—Dissolve 1 gm. in 10 mils of water, add a few milligrams of sodium chloride and 1 drop of indigo solution and mix the solution with 10 mils of sulphuric acid. A blue color should persist after the mixing.

Ammonium Salts.—Dissolve 1 gm. in 10 mils of water, add 5 mils of 27% sodium hydroxide solution, heat the mixture and test the vapors with moist red litmus paper. The paper should not turn blue.

ACID PICRIC**(Trinitrophenol; Picronic Acid)**

Mol. Wt. 229.08

Picric acid is usually in the form of pale yellow crystals. It is soluble in about 100 parts of water, in about 15 parts of alcohol and in about 20 parts of benzene. It melts between 121° and 123° C. (theory 122.5° C.).

Picric acid is liable to explode when quickly heated or subjected to percussion. In order to make safe its transportation it is usually mixed with 10% to 20% of water. Before making the tests of purity described below, added water should be removed by drying the chemical over sulphuric acid.

Picric acid is largely used as a general alkaloidal reagent and particularly in estimating cinchona alkaloids volumetrically; as a reagent for albumin, gelatin, hydrocyanic acid and gallic acid; in the colorimetric estimation of carbohydrates; in detecting creatin and guanin; in testing spermatic fluid; differentiating between benzene and benzoin; and in microscopy. It has also been recommended as a standard in acidimetry and iodometry, regarding which see Chem. Abst., 8, 2132, 3165 (1914).

Picric acid should be kept in well-stoppered bottles in a cool place, remote from fire.

Maximum Limits of Impurities

Substances Insoluble in Water	0.0000%
Alkali Picrates	0.0000%
Resins	0.0000%
Oxalic Acid ($\text{H}_2\text{C}_2\text{O}_4$)	0.1000%
Sulphuric Acid, Free and Combined (SO_3)	0.0750%
Inorganic Matter	0.1000%

Methods of Testing

Substances Insoluble in Water.—Dissolve 1 gm. of picric acid in 100 mls of water. The solution should be clear and no insoluble matter should remain.

Alkali Picrates.—Dissolve 1 gm. in 20 mls of benzene. The solution should be clear and free from insoluble matter.

Resins.—To 100 mls of the 1% aqueous solution of picric acid add 2 or 3 drops of diluted sulphuric acid and allow it to stand twelve to eighteen hours. No precipitate should form. Filter the liquid; no resinous matter should collect on the filter.

Oxalic Acid.—Dissolve 1 gm. in 100 mls of water, add calcium chloride solution and allow the mixture to stand two to three hours. No precipitate of calcium oxalate should form.

Sulphuric Acid, Free and Combined.—Evaporate a mixture of 2 gm. of picric acid and 10 mls of nitric acid (sp. gr. 1.40–1.42) to dryness on the steam-bath. Dissolve the residue in 100 mls of boiling water, add a few drops of nitric acid, allow to cool, then filter and to the filtrate add some barium nitrate solution. No turbidity should result.

Inorganic Matter.—Cautiously ignite 1 gm. of the picric acid. The residue should not weigh more than 0.001 gm.

Quantitative Method.—Dissolve about 1 gm., accurately weighed, in 100 mls of warm water and titrate with tenth-normal solution of sodium hydroxide, using phenolphthalein as indicator.

1 ml of tenth-normal NaOH = 0.022908 gm. of
 $C_6H_2OH(NO_2)_3$, log 35999.

ACID ROSOLIC

(Corallin; Aurin; Pararosolic Acid)

Rosolic acid ordinarily consists of a mixture of pseudo-rosolic acid, aurin, methyl aurin, and oxidized aurin. It is usually in the form of reddish-brown lumps having a greenish metallic luster. It is readily soluble in alcohol but almost insoluble in water. The test solution is prepared by dissolving 1 gm. in 100 mls of alcohol of about 50% strength.

As an indicator rosolic acid is serviceable in acidimetry and alkalimetry involving caustic alkalis and mineral acids, including sulphurous but excepting phosphoric acid. Carbon dioxide affects the indicator, but carbonates may be titrated in boiling solution. It is less satisfactory in estimating ammonia and in titrating in the presence of ammonium salts, and is not suitable for use with acetic acid and most other organic acids, though oxalic acid reacts sharply with it. It is used largely in gastric analysis and water analysis.

Rosolic acid should be stored in a cool place as otherwise it is liable to soften and become coherent.

Method of Testing

Add 2 or 3 drops of rosolic acid test solution to 100 mls of water and add 0.05 ml of tenth-normal solution of sodium hydroxide. A red color should be produced in the liquid which should be changed to yellow by the subsequent addition of 0.05 ml of tenth-normal hydrochloric acid solution.

ACID SALICYLIC

$\text{HC}_7\text{H}_5\text{O}_2$

Mol. Wt. 138.05

Salicylic acid usually occurs as fine, white, odorless, needle-like crystals or a crystalline powder. It is only slightly soluble in cold water (about 500 parts) but is soluble in about 2 parts of alcohol or ether and in about 30 parts of chloroform. It melts at about 157°C . (theory 159.05°C .).

The reagent is used principally for the detection of nitrites, acetone, titanium, formaldehyde, methyl alcohol and lactic acid and in nitrogen determinations by the Kjeldahl or Gunning method to include nitrate-nitrogen.

Salicylic acid is easily discolored by even traces of iron, such as may occur in dust. It should therefore be kept in well-stoppered containers.

Maximum Limits of Impurities

Non-volatile Matter	0.1000%
Organic Impurities	0.0000%
Chlorides (Cl)	0.0020%

Methods of Testing

Non-volatile Matter.—Ignite 1 gm. of the acid and weigh the residue. Not more than 0.001 gm. should be found.

Organic Impurities.—Dissolve 0.5 gm. in 10 mls of sulphuric acid. A dark brown color should not be produced although the liquid may become slightly yellowish.

Chlorides.—Dissolve 0.5 gm. in 10 mls of alcohol and add a few drops of nitric acid and silver nitrate solution. No reaction should be apparent.

ACID SILICOTUNGSTIC



Mol. Wt. 3312.72

Silicotungstic acid is usually in the form of white or slightly yellowish, deliquescent crystals. It is easily soluble in water and in alcohol. Upon ignition it yields not less than 85% nor more than 90% of residue. The determination of the residue upon ignition serves to distinguish the acid of the formula given above from others of different composition. The acid here described is that generally designated as silicotungstic acid in the literature descriptive of the analytical applications of the substance.

Silicotungstic acid is principally employed as an alkaloidal reagent, finding especial use in the quantitative determination of sparteine, atropine, nicotine, aconitine and brucine; also in estimating antipyrine and pyramidon. It is also used as a mordant for basic dyes.

The acid should be stored in glass-stoppered, air-tight containers in a cool place.

Maximum Limits of Impurities

Sulphates (SO ₄)	0.0100%
Chlorides (Cl)	0.0050%
Tungstates and Mercurous Salts	0.0000%
Heavy Metals	0.0000%

Methods of Testing

Sulphates.—Dissolve 0.5 gm. of silicotungstic acid in 10 mls of water and add barium chloride solution. No change should be apparent.

Chlorides.—Dissolve 0.5 gm. of the acid in 10 mls of water and add silver nitrate solution. Not more than a slight opalescence should be produced.

Tungstates and Mercurous Salts.—To 5 mls of a 10% solution of the silicotungstic acid add a few drops of hydrochloric acid. No change in the transparency of the solution should result.

Heavy Metals.—To the solution obtained in the preceding test add hydrogen sulphide water and allow the mixture to stand half an hour. No color or precipitate should develop.

Residue on Ignition.—Upon ignition to constant weight not less than 85% nor more than 90% of residue should be found.

ACID SUCCINIC



Mol. Wt. 118.07

Succinic acid occurs usually as colorless crystals, soluble in about 20 parts of cold and about 2 parts of boiling water; also soluble in about 10 parts of alcohol and in about 80 parts of ether. It melts between 182° and 183° C. (theory 185° C.) and contains at least 99.9% of $\text{H}_2\text{C}_4\text{H}_4\text{O}_4$. When heated to about 235° C. the acid boils with the evolution of white acrid fumes, at the same time decomposing to considerable extent into water and succinic anhydride.

The reagent is used chiefly in the separation of iron from aluminum, manganese, nickel, cobalt and zinc; as a reagent for calcium and albumin; and as a standard for preparing volumetric solutions. Regarding the last-named use see the article by Phelps and Hubbard, *Amer. J. Sci.*, **23**, (IV), 211 (1907); *abst.*, *J. Chem. Soc.*, **92**, II, 297 (1907) and *Chem. Abst.*, **1**, 1832 (1907).

Maximum Limits of Impurities

Non-volatile Matter	0.0250%
Sulphates (SO ₂)	0.0030%
Chlorides (Cl)	0.0025%
Ammonium Salts (NH ₄)	0.0020%
Heavy Metals	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the acid and weigh the residue. The weight should not exceed 0.0005 gm.

Sulphates.—Dissolve 1 gm. in 20 mls of water, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 2 to 3 mls of nitric acid and some silver nitrate solution. Not more than a very faint opalescence should be produced.

Ammonium Salts.—Heat 2 gm. of the acid with 10 mls of 27% sodium hydroxide solution and test the vapors with moist red litmus paper. The paper should not turn blue.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent.

Quantitative Method.—Dry the acid to constant weight in a desiccator over sulphuric acid, weigh out accurately 0.2 to 0.25 gm. of the dried material, dissolve it in water and titrate the solution with tenth-normal alkali solution, using phenolphthalein as indicator. At least 99.9% of H₂C₄H₄O₄ should be found.

1 mil of tenth-normal NaOH = 0.00590 gm. of H₂C₄H₄O₄,
log 77111.

ACID SULPHANILIC

C₆H₄(NH₂)(SO₃H)1:4 + 2H₂O Mol. Wt. 209.19

Sulphanilic acid is usually obtained as colorless, efflorescent crystals. It is soluble in about 160 parts of cold water,

more readily in hot water, but is almost insoluble in alcohol, ether or benzene. On heating it carbonizes without melting.

Sulphanilic acid is chiefly employed with alphanaphthylamine in the detection of nitrous acid in various materials, such as flour, drinking water, etc.; in urinalysis for the diazo reaction and the detection of bile pigments; in testing adrenalin solution, detecting betanaphthol and in microscopy.

The acid should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Inorganic Matter	0.0500%
Sulphates (SO_4)	0.0100%
Chlorides (Cl)	0.0050%

Methods of Testing

Inorganic Matter.—Ignite 2 gm. of sulphanilic acid. The residue should not weigh more than 0.001 gm.

Sulphates (Aniline Sulphate).—Dissolve 1 gm. in 25 mls of boiling water and add a few drops of barium chloride solution. No change should be apparent.

Chlorides (Aniline Hydrochloride).—Shake 1 gm. of sulphanilic acid with 20 mls of water, filter and add to the filtrate a few drops of nitric acid and silver nitrate solution. Not more than a slight opalescence should be produced.

ACID SULPHURIC, Sp. Gr. 1.84



Mol. Wt. 98.08

Sulphuric acid, sp. gr. 1.84, is a clear, colorless, oily liquid of specific gravity at least 1.84 and containing at least 94% of H_2SO_4 .

Among its many uses may be mentioned its employment as a precipitant for lead and barium, as a solvent of minerals, and in the Marsh test for arsenic, for which purpose it is well suited as it is readily obtained free from arsenic. Owing to the readiness with which it attacks organic matter

it is much used in work involving the destruction of organic compounds, as in nitrogen determinations, and in detecting organic impurities in various chemicals.

It should be stored in glass-stoppered bottles and should be well protected from contact with even traces of organic matter by which it is easily discolored. Owing to its great affinity for moisture the acid must be protected from deterioration in strength by keeping the bottles tightly stoppered and as far as possible avoiding undue exposure to air in storage and in handling. Paraffin, which is not attacked by the cold acid, may be used in sealing the containers.

Maximum Limits of Impurities

Non-volatile Matter	0.0005%
Nitric Acid (HNO ₃)	0.0010%
Selenium (Se)	0.0020%
Substances Oxidizable by Permanganate (SO ₂) ..	0.0010%
Hydrochloric Acid (HCl)	0.0003%
Lead (Pb)	0.0030%
Heavy Metals	0.0000%
Ammonium Salts (NH ₄)	0.0015%
Arsenic (As)	0.000003%

Methods of Testing

Non-volatile Matter (Calcium, Alkalis, etc.).—Evaporate 100 gm. of the acid and ignite and weigh the residue. It should weigh not more than 0.0005 gm.

Nitric Acid.—Dilute the acid with an equal volume of water and overlay 10 mls of the cooled liquid upon diphenylamine solution. A blue color should not appear at the zone of contact within one hour.

Selenium.—Overlay 3 mls of the acid with 9 mls of hydrochloric acid containing a few milligrams of sodium sulphite. A red color should not appear at the zone of contact.

Substances Oxidizable by Permanganate (Nitrous and Sulphurous Acids).—Add 0.05 mil of tenth-normal potassium permanganate solution to a mixture of 20 mls

of the acid and 60 mls of water. The pink color should persist for at least five minutes.

Hydrochloric Acid.—Dilute 20 mls of the acid with an equal volume of water and add silver nitrate solution. No turbidity should be visible on standing two to three hours.

Lead.—Dilute 10 mls of the acid with 50 mls of alcohol. No precipitate of lead sulphate should form on standing two hours.

Heavy Metals.—Dilute 10 mls of the acid with 100 mls of water and pass hydrogen sulphide gas into the mixture. No dark color or precipitate should be visible even after supersaturating the mixture with ammonia water.

Ammonium Salts.—Dilute 2 mls of the acid with 30 mls of water, make alkaline with about 15 mls of 27% sodium hydroxide solution and add 1 ml of Nessler's solution. No brownish-red color or precipitate should be produced, although a yellowish color or even a yellowish precipitate may be observed.

Arsenic.—Add 5 mls of nitric acid, sp. gr. 1.40–1.42, to 100 mls of the sulphuric acid and evaporate the mixture to about 10 mls. Dilute the residue with water to 50 mls, gradually introduce it into a Marsh apparatus and allow the action to proceed for two hours. No deposit of arsenic should be obtained.

Quantitative Method.—The percentage of sulphuric acid is conveniently obtained from the specific gravity by referring to the table on page 376. If desired the acid, after suitable dilution, may be titrated with standard alkali solution, using methyl orange as indicator. At least 94% of H_2SO_4 should be found.

1 ml of normal NaOH = 0.049038 gm. of H_2SO_4 ,
log 69053.

ACID SULPHURIC, 10%
(Diluted Sulphuric Acid)

 H_2SO_4

Mol. Wt. 98.08

Acid sulphuric, diluted, 10%, is a clear, colorless liquid of specific gravity about 1.069 and containing not less than 9.5% and not more than 10.5% of H_2SO_4 .

Diluted sulphuric acid is used as a precipitant for barium, strontium and lead, as a solvent, and for other purposes for which the concentrated acid is less suitable. It should be stored in glass-stoppered bottles.

Maximum Limits of Impurities

Non-volatile Matter	0.0005%
Nitric Acid (HNO_3)	0.0008%
Selenium (Se)	0.0040%
Substances Oxidizable by Permanganate (SO_2) ..	0.0003%
Hydrochloric Acid (HCl)	0.0001%
Lead (Pb)	0.0050%
Heavy Metals	0.0000%
Ammonium Salts (NH_4)	0.0002%
Arsenic (As)	0.000005%

Methods of Testing

Non-volatile Matter.—Evaporate 100 mls of the acid and ignite and weigh the residue. Not over 0.0005 gm. should be found.

Nitric Acid.—Overlay 10 mls of the acid upon diphenylamine solution. A blue color should not develop at the zone of contact within an hour.

Selenium.—Overlay 3 mls of the acid upon 3 mls of hydrochloric acid containing a few milligrams of sodium sulphite. A red color should not appear at the zone of contact.

Substances Oxidizable by Permanganate (Nitrous and Sulphurous Acids).—Add 0.1 mil of tenth-normal potassium permanganate solution to 100 mls of the acid. The pink color should persist for at least five minutes.

Hydrochloric Acid.—To 25 mls of the acid add silver nitrate solution. The mixture should remain clear.

Lead.—Dilute 10 mls of the acid with 50 mls of alcohol. No precipitate of lead sulphate should form on standing two hours.

Heavy Metals.—Pass hydrogen sulphide gas into 100 mls of the acid. No dark color should be visible even after adding an excess of ammonia water.

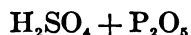
Ammonium Salts.—To 30 mls of the acid add sufficient 27% sodium hydroxide solution to make the mixture alkaline and then add 1 mil of Nessler's solution. No brownish-red color or precipitate should be produced, although a yellowish color or even a yellowish precipitate may be observed.

Arsenic.—Add 5 mls of nitric acid to 100 mls of diluted sulphuric acid and evaporate the mixture to fumes of sulphur trioxide. Dilute the residue with water to 50 mls, gradually introduce it into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be obtained.

Quantitative Method.—Titrate the acid with standard alkali solution, using methyl orange as indicator. Not less than 9.5% and not more than 10.5% of H_2SO_4 should be found.

1 mil of normal $\text{NaOH} = 0.049038$ gm. of H_2SO_4 , log 69053.

ACID SULPHURIC, WITH PHOSPHORIC ANHYDRIDE



Sulphuric acid with phosphoric anhydride is usually a clear, colorless or slightly yellowish or brownish, oily liquid, consisting of sulphuric acid to which phosphoric anhydride has been added until the mixture contains about 10% of free P_2O_5 .

The reagent is used in nitrogen determinations by the

Kjeldahl method for decomposing the more refractory organic substances. It should be stored in well-closed, glass stoppered bottles as it readily attracts moisture from the air and by so doing loses its content of free phosphoric anhydride.

Maximum Limits of Impurities

Nitric Acid (HNO_3)	0.0050%
Ammonium Salts (NH_3)	0.0020%

Methods of Testing

Nitric Acid.—Dilute 1 mil with 9 mils of water and overlay this mixture upon 5 mils of diphenylamine solution. No blue color should be produced at the plane of contact of the liquids.

Ammonium Salts.—Dilute 2 mils with 30 mils of water and 15% solution of potassium hydroxide (free from ammonium compounds) to alkalinity and then 10 to 15 drops of Nessler's solution. No brownish-red color or precipitate should result.

ACID SULPHURIC, FUMING

Fuming sulphuric acid is a clear, colorless, oily, fuming liquid. It contains not less than 13% and usually about 15% of free SO_3 . Fuming sulphuric acid of higher percentage is often desired for special purposes. Acids containing 50% to 60% or more of free SO_3 are obtainable; such acids are known commercially as "oleum."

The ordinary method of determining the strength of fuming sulphuric acid, does not take into account the presence of SO_2 , small amounts of which are nearly always present. If desired, the SO_2 content may be determined by titration with standard iodine solution and the acidimetric results corrected for the amount found. For details of procedure see Treadwell-Hall, *Analytical Chemistry*, 4th ed., II, 577 (1915).

Besides being suitable for Kjeldahl nitrogen determinations this acid is used in the examination of commercial

turpentine, in organic synthesis and gas analysis and as an oxidizing agent.

Fuming sulphuric acid should be stored in well-closed, glass-stoppered bottles. Contact with organic matter should be avoided in order to prevent discoloration or contamination of the acid. At somewhat below -10° C. crystallization often takes place in acid of the strength here described, hence a moderate temperature should be maintained.

Maximum Limits of Impurities

Non-volatile Matter	0.0100%
Nitric Acid (HNO_3)	0.0070%
Ammonium Salts (NH_4)	0.0020%
Halogens (Cl)	0.0025%
Lead (Pb)	0.0030%
Arsenic (As)	0.0001%

Methods of Testing

Non-volatile Matter.—Evaporate and ignite 5 gm. of the acid. Not more than 0.0005 gm. of residue should remain.

Nitric Acid.—Dilute 1 mil with 9 mils of water and overlay the mixture upon 5 mils of diphenylamine solution. A blue color should not be visible at the plane of contact of the two liquids.

Ammonium Salts.—Add 2 mils of the acid to 40 mils of water, make alkaline with 27% sodium hydroxide solution and add 1 mil of Nessler's solution. A brownish-red color or precipitate should not be produced though a yellowish color or precipitate may result.

Halogens.—Add 1 mil of the acid to 25 mils of water and afterwards add silver nitrate solution. At most a slight opalescence should be produced.

Lead.—Add 10 mils of the acid by drops to 50 mils of alcohol and allow the mixture to stand for two hours. At the end of this time no precipitate of lead sulphate should be visible.

Arsenic.—Dilute 10 mls of the acid with water to 100 mls and introduce the mixture into a Marsh apparatus, After the test has proceeded for one hour no deposit of arsenic should be visible in the reduction tube.

Quantitative Method.*—Cautiously run the weighed sample† of fuming sulphuric acid into 50 to 100 mls of water and titrate with standard alkali solution, using methyl orange as indicator. At least 84% of total SO_3 should be found, corresponding to at least 13% of free SO_3 .‡

1 mil of normal $\text{NaOH} = 0.040035$ gm. of SO_3 , log 60245

ACID SULPHURIC, FUMING

For Nitrogen Determination

Fuming sulphuric acid for nitrogen determination is an oily, fuming liquid, sometimes slightly colored or cloudy. It contains not less than 13% and usually about 15% of free SO_3 .

This acid may contain traces of lead and other impurities which however do not render it unfit for nitrogen determinations by the Kjeldahl method for which it is especially intended. It should be stored in well-closed, glass-stoppered bottles.

Maximum Limit of Impurity

Nitrogen 0.0010%

* The approximate strength of fuming sulphuric acid, particularly for technical purposes, may be easily determined from its specific gravity [see Gavelle, *Mon. Sci.* 3, 301 (1913); *abst.*, *Chem. Abst.*, 7, 2527 (1913)] or by titration with water [Parker, *J. Soc. Chem. Ind.*, 36, 692 (1917); *abst.*, *Chem. Abst.*, 11, 3007 (1917)].

† For weighing samples of the acid the Lunge-Rey bulb pipette is convenient. Other forms of apparatus more complicated but claimed to be more reliable are described by Finch [*Chem. Abst.*, 4, 1828 (1910)] and by Vernon [*Chem. Abst.*, 4, 2893 (1910)].

‡ The percentage of free SO_3 may be readily obtained by referring to tables on page 377.

Methods of Testing

Nitrogen.—Dilute 30 mls of the acid with 100 mls of water, add 3 gm. of zinc dust and allow the mixture to stand with frequent stirring until the evolution of hydrogen has ceased. Then add 27% sodium hydroxide solution until strongly alkaline and distil, collecting the distillate in a measured quantity of tenth-normal acid solution. Titrate the excess acid with tenth-normal alkali solution, using methyl orange as indicator. Not more than 0.4 mil of tenth-normal acid solution should be neutralized by the distillate.

The nitrogen content of the reagents used in this test may be determined, if necessary for correcting the result, by a blank test upon sulphuric acid known to be free from nitrogen; or, if such acid is not available, another test may be made upon the acid in the same manner and with the same quantities of reagents but with double the amount (60 mls) of the fuming sulphuric acid. The difference between the quantities of tenth-normal acid consumed in the two tests is a measure of the nitrogen in 30 mls of the fuming acid.

Quantitative Method.—This is to be carried out as directed under Acid Sulphuric, Fuming. At least 84% of total SO_3 should be found, corresponding to at least 13% of free SO_3 .

ACID SULPHURIC, FUMING, WITH PHOSPHORIC ANHYDRIDE

This reagent consists of fuming sulphuric acid containing in solution about 10% of P_2O_5 . It has the general appearance and properties of Acid Sulphuric, Fuming, For Nitrogen Determination, and the precautions to be observed in storing it are the same as those stated under that subject. It is likewise intended for nitrogen determination.

Maximum Limit of Impurity

Nitrogen (N) 0.0010%

Method of Testing

Nitrogen.—This test is to be carried out as described under Acid Sulphuric, Fuming, For Nitrogen Determinations. The distillate obtained should not neutralize more than 0.4 mil of tenth-normal hydrochloric acid solution.

ACID SULPHUROUS
 $\text{SO}_2 + \text{Aq.}$

Mol. Wt. 64.06

Sulphurous acid is a clear, colorless liquid of specific gravity about 1.030 and containing at least 5.5% of SO_2 . When saturated with sulphur dioxide at 15° C. it contains about 10%, but easily loses the gas by volatilization, to some extent also by oxidation, sulphuric acid being nearly always present. The acid prepared in cold weather is generally found of higher percentage than that produced in hot weather, due to the volatility of the gas.

Sulphurous acid is mainly used as a reducing agent; also in the determination of copper with potassium sulphocyanate and as a reagent for pine oil.

The acid should be stored in tightly-closed, glass-stoppered bottles in a cool place; extreme cold should be avoided as at a temperature somewhat below 0° C. sulphurous acid congeals. It should also be protected from light which exerts upon it a reducing action, causing slow deposition of sulphur.

Maximum Limit of Impurity

Non-volatile Matter 0.0050%

Methods of Testing

Non-volatile Matter.—Evaporate 10 mils of sulphurous acid and ignite and weigh the residue. Not more than 0.0005 gm. should be found.

Quantitative Method.—Introduce the sulphurous acid into a measured excess of tenth-normal iodine solution, previously tared, and weigh the mixture to obtain the weight of the sample taken. Titrate the excess iodine with

tenth-normal sodium thiosulphate solution. At least 5.5% of SO_2 should be found.

1 mil of tenth-normal I = 0.003203 gm. of SO_2 , log 50556.

ACID TANNIC

(Gallotannic Acid; Tannin)

Tannic acid is usually in the form of a yellowish powder or lustrous scales, soluble in about 1 part or less of water, alcohol or warm glycerin; almost insoluble in chloroform or ether. Its solutions are dextrorotary.

Chief among its many uses are the detection of iron, albumin, alkaloids and gelatine; the detection of artificial coloring in wine and beer; testing arecoline, caffeine, homatropine, blood, aniline dyes, etc.; and determining the effective value of hide powder and solutions used in estimating tannin. Its 10% solution is employed as a test solution in the U. S. Pharmacopœia, 9th Revision.

Tannic acid should be stored in air-tight containers in a cool place protected from light since air and light gradually discolor it. Solutions spoil easily and should be freshly prepared when required.

Maximum Limits of Impurities

Non-volatile Matter	0.1000%
Zinc (Zn)	0.0050%
Sugar, Dextrin	0.0000%
Excess Water	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 5 gm. of tannic acid. Not more than 0.005 gm. of ash should remain.

Zinc.—Dissolve the ash obtained in the above test in 2 mls of 36% acetic acid, dilute the solution with 8 mls of water, filter and add hydrogen sulphide water to the filtrate. Not more than a slight opalescence should be produced.

Sugar, Dextrin.—Dissolve 2 gm. of tannic acid in 10 mls of water, add 10 mls of alcohol and allow to stand one

hour. The mixture should remain clear. Then add 5 mls of ether. No turbidity should be produced.

Excess Water.—Dry tannic acid at 100° C. until the weight is constant. The loss should not exceed 12%.

Note.—Regarding the quantitative determination of tannic acid see Allen's Commercial Organic Analysis, 4th ed., 5, 61 (1911) and 9, 389, 394, 404 (1917).

ACID TARTARIC



Mol. Wt. 150.07

Tartaric acid for reagent use is usually in the form of clear, colorless crystals, soluble in less than 1 part of water and in about 4 parts of alcohol. It contains at least 99.5% of $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$.

The tartaric acid of the market often contains traces of lead. For the medicinal acid a limit of 20 parts per million has been established by the U. S. Pharmacopœia, 9th Revision, and a colorimetric method of determining this quantity of lead is there given.

Tartaric acid is used mainly in the detection of potassium and resorcinol, in the separation of iron and aluminum oxides and in the estimation of iron and antimony.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0100%
Calcium (Ca)	0.0100%
Heavy Metals	0.0000%
Inorganic Matter	0.0500%
Oxalic Acid ($\text{H}_2\text{C}_2\text{O}_4$)	0.0200%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 20 mls of water and add barium chloride solution. The liquid should remain clear.

Calcium.—Dissolve 1 gm. in 20 mls of water and add ammonium oxalate solution. No change should be apparent.

Heavy Metals.—Dissolve 10 gm. of tartaric acid in 40 mls of water and divide the solution into two equal por-

tions. To one add hydrogen sulphide water. No color or precipitate should be produced. To the other portion add ammonia water in slight excess (12 to 15 mls) followed by hydrogen sulphide water. No brown color should develop.

Inorganic Matter.—Ignite 5 gm. of tartaric acid and weigh the ash. The weight should not exceed 0.0025 gm.

Oxalic Acid.—Dissolve 4 gm. of tartaric acid in 20 mls of water, add ammonia water until the solution is only slightly acid and then calcium sulphate solution. No change should be apparent.

Quantitative Method.—Dissolve the acid in water and titrate the solution with standard alkali solution, using phenolphthalein as indicator. At least 99.5% of $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ should be found.

1 mil of normal $\text{NaOH} = 0.075035$ gm. of $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$,
log 87526.

AGAR

(Agar-agar)

Agar usually occurs as bundles of thin, translucent, membranous, agglutinated, yellowish-white shreds or a pale buff, coarse powder having a mucilaginous taste and not more than a slight odor. It is insoluble in cold water but slowly dissolves in hot water, the 1% solution yielding a stiff jelly on cooling.

Agar is used chiefly in the preparation of bacteriological culture media.

Maximum Limits of Impurities

Ash	5.0%
Gelatine	1.5%
Starch	1.5%

Methods of Testing

Ash.—Ignite the agar until carbonaceous matter is entirely removed. The residue should not exceed 5% of the weight of the agar taken.

Gelatine.—Add solution of tannic acid to a clear, cold solution of 0.1 gm. of agar in 100 mls of water. No precipitate should form nor should the mixture become markedly opalescent.

Starch.—Dissolve 0.1 gm. of agar in 100 mls of water, cool the solution and add a few drops of iodine solution. No blue color should develop.

ALCOHOL, 95% (Ethylic Alcohol)

C_2H_5OH

Mol. Wt. 46.05

Alcohol, 95%, is a clear, colorless, volatile, inflammable liquid having a slight, characteristic odor. It is very hygroscopic and is miscible with water in all proportions; also miscible with ether and chloroform. Alcohol, 95%, boils at about 78° C. and has a specific gravity not above 0.816 at 60°/60° F. It contains at least 95% by volume, corresponding to 92.4% by weight, of C_2H_5OH . The reagent is neutral to litmus paper, previously moistened with water.

Alcohol is employed chiefly as a solvent; as a vehicle for the application of other reagents; as a physical precipitant for various substances; as a reducing agent; and for the formation of esters in synthesis and analysis.

Alcohol should be stored in well-stoppered bottles, remote from fire. If kept in cork stoppered containers for a length of time the alcohol is likely to take up traces of organic matter from the stoppers which may interfere with the test for organic impurities.

Maximum Limits of Impurities

Residue on Evaporation.....	0.0010%
Fusel Oil	0.0000%
Molasses Alcohol	0.0000%
Aldehyde	0.0000%
Acetone	0.0200%
Furfural	0.0001%
Heavy Metals	0.0000%
Tannin	0.0002%
Organic Impurities	0.0000%

Methods of Testing

Residue on Evaporation.—Evaporate 60 mls on the steam-bath and weigh the residue. Not more than 0.0005 gm. should be found.

Fusel Oil.—Mix 10 mls with 30 mls of water. No color or turbidity should be apparent nor should a foreign odor develop.

Mix 10 mls with 5 mls of water and 1 ml of glycerin and allow the mixture to evaporate spontaneously from a piece of clean, odorless blotting paper. No foreign odor should be perceptible when the last traces of alcohol leave the paper.

Rub a few drops of the alcohol between the hands. No unpleasant odor should be noticeable.

Molasses Alcohol.—Overlay 5 mls upon 5 mls of sulphuric acid and allow it to stand one hour. No rose-red color should be produced at the plane of contact of the liquids.

Aldehyde.—Mix 10 mls with 10 mls of water, add 2 mls of ammoniacal silver nitrate solution and allow to stand twelve to eighteen hours in a dark place. No color or turbidity should develop.

Acetone.—Shake for one minute a mixture of 6 mls of barium hydroxide solution, 0.2 ml of 5% mercuric chloride solution and 2 mls of alcohol. Filter and to the filtrate add ammonium sulphide solution. No dark color should be produced.

Furfural.—Mix 10 mls of alcohol with 1 drop of aniline and 5 drops of 36% acetic acid, and allow the mixture to stand one hour. No red color should develop.

Heavy Metals.—To 10 mls of alcohol add 5 mls of hydrogen sulphide water. No change should be apparent.

Tannin.—To 10 mls of alcohol add 5 mls of 5% solution of sodium hydroxide. No color should be produced.

Organic Impurities.—To 10 mls of alcohol, cooled to about 15° C., add 0.05 ml of tenth-normal potassium per-

manganate solution and allow to stand five minutes. The pink color should not be entirely discharged.

ALCOHOL, ABSOLUTE

(Absolute Ethylic Alcohol; Dehydrated Alcohol)

C_2H_5OH

Mol. Wt. 46.02

Absolute alcohol has the same general properties, physical appearance and miscibility as alcohol, 95%. It boils at about 78° C. (theory 78.4° C.) and has a specific gravity not above 0.797 at 60°/60° F. It contains at least 99.4% by volume, corresponding to 99.0% by weight, of C_2H_5OH . It is neutral to moistened litmus paper.

Absolute alcohol is used in the same way as alcohol, 95%, and also for special purposes requiring greater freedom from water. The reagent should be stored with the same precautions as alcohol, 95%.

Maximum Limits of Impurities

Residue on Evaporation	0.0010%
Fusel Oil	0.0000%
Molasses Alcohol	0.0000%
Aldehyde	0.0000%
Acetone	0.0200%
Furfural	0.0001%
Heavy Metals	0.0000%
Tannin	0.0002%
Organic Impurities	0.0000%

Methods of Testing

The methods of testing Alcohol, Absolute, are the same as those given under Alcohol, 95%. The results should be as there stated.

ALCOHOL, AMYLIC

$C_5H_{11}OH$

Mol. Wt. 88.12

Amylic alcohol is a clear, colorless liquid having a characteristic penetrating odor. It has a specific gravity of 0.812 to 0.815 (theory 0.8169 at 18° C.) and boils at 130° to

132° C. (theory 128.0°). Amyl alcohol is only slightly soluble in water but is miscible with alcohol, ether and benzene. It is neutral to moistened litmus paper.

Amylic alcohol is used chiefly as a solvent, especially in alkaloidal analysis.

Maximum Limits of Impurities

Residue on Evaporation	0.0050%
Organic Impurities	0.0000%

Methods of Testing

Residue on Evaporation.—Evaporate 10 gm. of amylic alcohol on the steam-bath and weigh the residue. The weight should not exceed 0.0005 gm.

Organic Impurities (Furfural, etc.).—Shake 5 mls with 5 mls of sulphuric acid. Not more than a slight yellow or reddish color should be produced.

Shake 5 mls with 5 mls of 30% solution of potassium hydroxide. The amylic alcohol should not acquire a color.

ALCOHOL, AMYLIC, FOR FAT DETERMINATION

$C_5H_{11}OH$

Mol. Wt. 88.12

Amyl alcohol for fat determination is a clear, colorless liquid having a specific gravity of 0.815 to 0.818 (theory 0.8169 at 18° C.) and boiling at 124 to 130° C. (theory 128.0° C.).

The reagent is especially intended for use in the determination of fat in milk with the Gerber butyrometer. Regarding this method see Allen's Commercial Organic Analysis, 4th ed., 8, 152 (1913).

Method of Testing

Shake 1 mil of the amylic alcohol with 10 mls of sulphuric acid (sp. gr. 1.820 to 1.825) and 11 mls of water in a Gerber butyrometer, centrifuge the mixture for two or three minutes and then allow it to stand twenty-four hours. No oily layer should separate.

ALCOHOL, METHYLIC**CH₃OH****Mol. Wt. 32.04**

Methyl alcohol for use as a reagent is a clear, colorless liquid of specific gravity not higher than 0.799 and containing at least 99.4% by volume of CH₃OH. It is clearly miscible with water, alcohol and ether, and boils at about 67° C. (theory 66.78° C.). It is neutral to litmus paper.

Methyl alcohol is used largely as a solvent, especially in preparing solutions for imbedding and staining in bacteriology, pathology, etc., and for many such purposes is considered superior to ethyl alcohol. It is used in the detection of skatol, salicylic acid and boric acid, and in the estimation of the latter; also in preparing pure, anhydrous dextrose.

Methyl alcohol is easily volatile and inflammable and when exposed to direct sunlight gradually acquires a content of formaldehyde. It should therefore be stored in well-stoppered containers in a cool place and should be protected from sunlight.

Maximum Limits of Impurities

Non-volatile Matter	0.0020%
Acetone	0.0150%
Ethyl Alcohol	1.0000%
Chloroform	0.0100%
Aldehydes	0.0000%
Empyreuma	0.0000%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 30 mls on a water bath. Not more than 0.0005 gm. of residue should remain.

Acetone and Ethyl Alcohol.—Mix 5 mls of methyl alcohol with 50 mls of double-normal sodium hydroxide solution and add 25 mls of double-normal iodine solution with repeated shaking. No turbidity or precipitate should be visible, and after warming to 60°–70° C. for half an hour no odor of iodoform should be perceptible.

Chloroform.—Mix 10 mls with an equal volume of alcoholic 10% solution of potassium hydroxide, add about 0.1 mil of aniline and heat the mixture to boiling. No odor of isonitrile should develop.

Aldehydes.—Mix 10 mls with an equal volume of 27% solution of sodium hydroxide. The mixture should be colorless.

Empyreumatic Substances.—Add 5 mls of sulphuric acid by drops to 5 mls of the alcohol, kept cold and well agitated during the addition. The mixture should not be more than slightly yellowish in color.

Substances Reducing Permanganate.—Add 0.05 mil of tenth-normal solution of potassium permanganate to 10 mls of methyl alcohol and keep the mixture at about 15° C. for ten minutes. The pink color should not be entirely discharged at the end of that time.

Quantitative Methods.—The specific gravity serves as a means of determining the strength of methyl alcohol. At least 99.4% by volume, of CH_3OH should be found.

Tables of specific gravities and percentages may be found on page 379. Compare Allen's Commercial Organic Analysis, 4th ed., 1, 86 (1909). Consult the same work, 1, 91 (1911), and 9, 1 (1917) regarding the determination of methyl alcohol by other means.

ALPHANAPHTHOL

$\text{C}_{10}\text{H}_7\text{OH}$

Mol. Wt. 144.11

Alphanaphthol usually occurs as colorless, lustrous crystals, having a phenolic odor. It is only slightly soluble in water but is easily soluble in alcohol or ether. The reagent melts at about 94° C. (theory 94.2° C.).

Alphanaphthol is used mainly for the detection of sugar, lignin, hydrated chloral, chloroform, nitrous acid, free acids and acid salts; as a reagent for free hydrochloric acid in gastric juice, carbohydrates in urine, cotton in woollens and foreign carbohydrates in mannite.

The reagent should be stored in well-stoppered bottles, protected from light.

Maximum Limits of Impurities

Organic Acids	0.0000%
Organic Substances Insoluble in Sodium Hydroxide Solution	0.0000%
Inorganic Matter	0.0500%

Methods of Testing

Organic Acids.—Shake 1 gm. with 100 mls of water, filter and test the filtrate with blue litmus paper. The paper should not be reddened.

Organic Substances Insoluble in Sodium Hydroxide Solution.—Dissolve 1 gm. in 10 mls of 10% sodium hydroxide solution. The solution should be clear and free from insoluble matter.

Inorganic Matter.—Ignite 2 gm. of alphanaphthol and weigh the residue. The weight should not exceed 0.001 gm.

ALPHANAPHTHYLAMINE HYDROCHLORIDE

$C_{10}H_7NH_2 \cdot HCl$

Mol. Wt. 179.60

Alphanaphthylamine hydrochloride usually occurs as a white, crystalline powder having a characteristic, disagreeable odor. It is soluble in about 25 parts of water and is readily soluble in alcohol and in ether.

On exposure to the air and light the salt acquires a bluish color which, if only slight, does not interfere materially with its ordinary uses. Its solutions are turned blue by the air or by oxidizing agents such as ferric chloride or chromic acid. A discolored solution may be decolorized by shaking with a little zinc dust.

The reagent is used chiefly in the detection and determination of nitrites, especially small quantities such as occur in water, foods or in bacterial cultures. It should be stored in well-stoppered, amber-colored bottles.

Maximum Limits of Impurities

Insoluble Matter	Trace
Non-volatile Matter	0.0500%

Methods of Testing

Insoluble Matter.—Dissolve 0.5 gm. in 20 mls of warm water. The solution should be colorless and not more than opalescent.

Non-volatile Matter.—Ignite 2 gm. of alphanaphthylamine hydrochloride. Not more than 0.001 gm. of residue should remain.

AMMONIA WATER

$\text{NH}_3 + \text{Aq.}$

Mol. Wt. $\text{NH}_3 = 17.03$

Ammonia water is a clear, colorless liquid of extremely pungent odor and consists of a solution of ammonia gas, NH_3 , in water. For analytical purposes three different strengths of ammonia water are ordinarily used, containing 28%, 20% and 10% of NH_3 , respectively.

Solutions of ammonia attack containers, especially those of glass, dissolving a portion which appears in the determination of their content of non-volatile matter. A small residue is therefore nearly always obtained in making this test.

Ammonia water is extensively used, principally as an alkalizing agent, as a group precipitant and solvent in analysis and for preparing volumetric solutions employed in acidimetry.

The reagent should be stored in tightly-stoppered bottles, in a cool place. Plenty of air space should be left in the containers to allow for expansion with changes of temperature.

I**AMMONIA WATER, 28%****(Stronger Ammonia Water)**

Ammonia water, 28%, has a specific gravity of about 0.90 and contains at least 27%, and usually about 28% of NH_3 .

Maximum Limits of Impurities

Non-volatile Matter	0.0150%
Carbon Dioxide (CO ₂)	0.0200%
Pyridine	Trace
Tar Bases	0.0000%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0003%
Sulphates (SO ₄)	0.0025%
Sulphides (S)	0.0010%
Calcium (Ca)	0.0020%
Magnesium (Mg)	0.0010%
Phosphates (P ₂ O ₅)	0.0015%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 15 mls of the ammonia water on the steam-bath. The residue should not weigh more than 0.002 gm.

Carbon Dioxide.—Mix 5 mls of the ammonia water with 20 mls of lime water and heat to boiling. The liquid should not become more than slightly turbid.

Pyridine.—Dilute 15 mls of ammonia water with 20 mls of water and nearly neutralize with sulphuric acid of about 25% strength. Not more than a slight odor of pyridine should be perceptible.

Tar Bases (Aniline, Pyridine, Pyrrol, etc.).—Evaporate on the steam-bath a mixture of 3.5 mls of ammonia water and 25 mls of nitric acid. The residue should have a pure white color.

Heavy Metals.—Dilute 2 mls of ammonia water with 20 mls of water and add a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Chlorides.—Dilute 3.5 mls of ammonia water with 20 mls of water. Acidify with 10 mls of nitric acid and add silver nitrate solution. No change in the transparency of the liquid should be apparent.

Sulphates.—Dilute 3.5 mls of ammonia water with 15 mls of water, acidulate with 5 mls of hydrochloric acid,

add barium chloride solution, and allow to stand twelve to eighteen hours. No precipitate should form.

Sulphides.—Add a few drops of ammoniacal lead acetate solution to 3.5 mls of the ammonia water. No yellow or brown color should be produced nor should a dark precipitate form.

Calcium.—Dilute 3.5 mls with 20 mls of water and add ammonium oxalate solution. The liquid should not become turbid.

Magnesium.—Dilute 7 mls with 15 mls of water, add ammonium phosphate solution and allow it to stand two hours. No precipitate should form.

Phosphates.—To 7 mls add 30 mls of nitric acid and 25 mls of ammonium molybdate solution and allow it to stand two hours at about 40° C. No yellow precipitate should form.

Substances Reducing Permanganate.—Dilute 3.5 mls of ammonia water with 5 mls of water, add 50 mls of 10% sulphuric acid and 0.1 ml of tenth-normal potassium permanganate solution, heat the mixture to boiling and boil for five minutes. The pink color should not be entirely discharged. Account must be taken of any reduction of the permanganate by the water and the sulphuric acid.

Quantitative Method.—Titrate a weighed sample of the ammonia water, suitably diluted, with standard acid solution, using methyl orange as indicator. At least 27% of NH_3 should be found.

1 ml normal HCl = 0.01703 gm. of NH_3 , log 23121.

The strength of the ammonia water may also be ascertained from its specific gravity by referring to the tables of specific gravity and per cent of ammonia solutions on page 378.

II

AMMONIA WATER, 20%

Ammonia water, 20%, has a specific gravity of about 0.925 and contains not less than 19% and usually about 20% of NH_3 .

Maximum Limits of Impurities

Non-volatile Matter	0.0108%
Carbon Dioxide (CO_2)	0.0150%
Pyridine	Trace
Tar Bases	0.0000%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0002%
Sulphates (SO_4)	0.0017%
Sulphides (S)	0.0007%
Calcium (Ca)	0.0015%
Magnesium (Mg)	0.0007%
Phosphates (P_2O_5)	0.0010%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Carry out the tests described under Ammonia Water, 28%; but instead of using 2 mls, 3.5 mls, 5 mls, 7 mls and 15 mls as there directed, use 3 mls, 5 mls, 7 mls, 10 mls and 20 mls respectively of the Ammonia Water, 20%. The results should be as stated under Ammonia Water, 28%.

III

AMMONIA WATER, 10%

Ammonia water, 10%, has a specific gravity of about 0.96 and contains not less than 9.5%, and usually about 10% of NH_3 .

Maximum Limits of Impurities

Non-volatile Matter	0.0052%
Carbon Dioxide (CO_2)	0.0100%
Pyridine	Trace
Tar Bases	0.0000%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0001%

Sulphates (SO_4)	0.0008%
Sulphides (S)	0.0004%
Calcium (Ca)	0.0008%
Magnesium (Mg)	0.0004%
Phosphates (P_2O_5)	0.0005%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Carry out the tests described under Ammonia Water, 28%; but instead of using 2 mls, 3.5 mls, 5 mls, 7 mls, and 15 mls as there directed, use 5 mls, 10 mls, 15 mls, 20 mls and 40 mls respectively of the Ammonia Water 10%. The results should be as stated under Ammonia Water, 28%.

AMMONIUM ACETATE

$\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$

Mol. Wt. 77.07

Ammonium acetate generally occurs as colorless crystals, sometimes as white crystalline masses, usually having a slight, acetous odor. The salt is easily soluble in water (less than 1 part) and in alcohol and is hygroscopic.

Ammonium acetate is employed principally in the determination of lead and iron and in separating the sulphates of lead and calcium from each other and from those of barium and strontium.

The reagent should be stored in tightly-stoppered containers.

Maximum Limits of Impurities

Non-volatile Matter	0.0100%
Chlorides (Cl)	0.0010%
Sulphates (SO_4)	0.0100%
Heavy Metals	0.0000%
Calcium (Ca)	0.0040%

Methods of Testing

Non-volatile Matter.—Ignite 10 gm. of the ammonium acetate. Not more than 0.001 gm. of residue should be left.

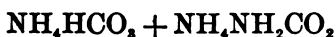
Chlorides.—Dissolve 1 gm. in 20 mls of water and add 5 mls of nitric acid and some silver nitrate solution. The mixture should be free from opalescence.

Sulphates.—Dissolve 5 gm. in 20 mls of water, acidulate with 1 mil of hydrochloric acid, add barium chloride solution and allow the mixture to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Heavy Metals.—Dissolve 5 gm. in 100 mls of water and add hydrogen sulphide water, followed by ammonia water in slight excess. No dark color or precipitate should be produced by either reagent.

Calcium.—Dissolve 5 gm. in 100 mls of water and add ammonium oxalate solution. No turbidity should be produced.

AMMONIUM CARBONATE



Ammonium carbonate usually occurs as white, hard, translucent lumps. It consists of a mixture of ammonium bicarbonate and ammonium carbamate in somewhat varying proportions and contains not less than 30% nor more than 32% of NH_3 . It is soluble in about 5 parts of water but only partly soluble in alcohol.

On exposure to the air ammonium carbonate loses ammonia and carbon dioxide and is finally converted into porous, friable masses or a white powder. Alcohol dissolves out the carbamate leaving the bicarbonate, and on dissolving the reagent in water the carbamate is converted into the carbonate, $(\text{NH}_4)_2\text{CO}_3$.

Ammonium carbonate is used in analysis, particularly as a group reagent for the alkali earths and for separating them from magnesium and the alkalis; for the separation of arsenic from antimony, magnesium from lithium, and chlorine from bromine and iodine.

It should be stored in well-stoppered, air-tight containers in a cool place. Glass-stoppered bottles are preferable.

Maximum Limits of Impurities

Non-volatile Matter	0.0050%
Sulphates (SO_4)	0.0009%

Chlorides (Cl)	0.0005%
Thiosulphates [(NH ₄) ₂ S ₂ O ₃]	0.0008%
Phosphates (P ₂ O ₅)	0.0050%
Sulphocyanates (CNS)	0.0250%
Heavy Metals	0.0000%
Tar Bases	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 10 gm. of ammonium carbonate and weigh the residue. Not more than 0.0005 gm. should be found.

Sulphates.—Dissolve 20 gm. of ammonium carbonate in a mixture of 150 mls of water and 40 mls of hydrochloric acid, boil the liquid, add barium chloride solution and let the mixture stand about fifteen hours. Any precipitate formed should not weigh over 0.0005 gm.

Chlorides and Thiosulphates.—Dissolve 2 gm. in 20 mls of water, add 10 mls of nitric acid and some silver nitrate solution. The liquid should not appear changed.

Phosphates.—Dissolve 4 gm. in 20 mls of water with the aid of 10 mls of nitric acid, add 20 mls of ammonium molybdate solution and keep the mixture at about 40° C. for fifteen minutes. No yellow precipitate should form.

Sulphocyanates.—Dissolve 5 gm. in 20 mls of water with the aid of 5 mls of hydrochloric acid and add a drop of ferric chloride solution. The mixture should not be reddened.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water and add a few drops of ammonium sulphide solution. No green or brown color should develop.

Dissolve 2 gm. in 20 mls of water, acidulate the solution with 4 mls of hydrochloric acid and add 10 mls of hydrogen sulphide water. The mixture should not be colored.

Tar Bases (Aniline, etc.).—Dissolve 1 gm. in 5 mls of nitric acid and evaporate the solution to dryness on the steam-bath. The residue should be white.

Quantitative Method.—Select and weigh translucent pieces of ammonium carbonate,* dissolve them in an excess of standard acid solution, previously diluted with an equal volume of water and titrate the excess acid with standard alkali solution, using methyl orange as indicator. From 30% to 32% of NH_3 should be found.

1 mil of normal HCl = 0.01703 gm. of NH_3 , log 23121.

AMMONIUM CHLORIDE

NH_4Cl

Mol. Wt. 53.50

Ammonium chloride is usually a white, granular powder, readily soluble in about 3 parts of water and in about 80 parts of alcohol. When heated it sublimes without melting. When kept cold, its aqueous solutions are generally neutral, but on warming, a little ammonia is lost and a slightly acid reaction is acquired.

Ammonium chloride, by virtue of its solvent action upon various hydroxides and carbonates, is mainly used in the separation of the iron and zinc groups of metals and of magnesium from barium, strontium and calcium. It is also used for transposing various metallic oxides and salts to the chlorides; to aid in the removal of volatile chlorides by sublimation; as a precipitant for aluminum, platinum and iridium; and in the estimation of uric acid. It is a constituent of magnesia mixture.

Ammonium chloride is slightly hygroscopic and should therefore be kept in well-stoppered bottles.

Maximum Limits of Impurities

Non-volatile Matter	0.0100%
Phosphates (P_2O_5)	0.0010%
Arsenates (As_2O_5)	0.0050%
Sulphates (SO_4)	0.0050%
Sulphocyanates (CNS)	0.0250%
Tar Bases	0.0000%
Heavy Metals	0.0000%

* In order to avoid loss of ammonia while weighing, a weighing-bottle containing enough water to cover the ammonium carbonate is tared, the sample added and, after weighing again, the whole contents are washed into the standard acid solution.

Methods of Testing

Non-volatile Matter.—Ignite 10 gm. of ammonium chloride. Not more than 0.001 gm. of residue should be left.

Phosphates and Arsenates.—Dissolve 5 gm. in 20 mls of water, add 3 mls of magnesia mixture and 10 mls of ammonia water and allow the mixture to stand about fifteen hours. No precipitate should form.

Sulphates.—Dissolve 5 gm. in 100 mls of water, add 5 mls of hydrochloric acid and some barium chloride solution and allow the mixture to stand about fifteen hours. No precipitate should form.

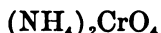
Sulphocyanates.—Dissolve 5 gm. in 25 mls of water and add a drop of ferric chloride solution. A red color should not be produced.

Tar Bases (Aniline, etc.).—Mix 1 gm. of ammonium chloride with 5 mls of nitric acid and evaporate the mixture to dryness on the steam-bath. The residue should be white.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water, add a few drops of hydrochloric acid and 10 mls of hydrogen sulphide water. The mixture should be clear and colorless. Then add a slight excess of ammonia water. No green or brown color should be produced.

AMMONIUM CHROMATE

(Neutral Ammonium Chromate)



Mol. Wt. 152.08

Ammonium chromate is usually in the form of yellow, needle-shaped crystals, soluble in about 5 parts of water, yielding a solution alkaline to litmus. The reagent contains at least 99.5% of $(\text{NH}_4)_2\text{CrO}_4$.

The salt on long exposure to the air or at a gentle heat gradually loses ammonia and is converted to the dichromate. Its solutions undergo a like change upon evaporation, making purification difficult; but a pure salt may be

obtained by crystallization in an atmosphere of ammonia. On ignition the salt is converted to the green chromic oxide, Cr_2O_3 .

Ammonium chromate is used chiefly in separating the alkaline earth metals; in detecting lead, mercury, silver and barium; and for many of the uses to which potassium chromate is put.

The reagent should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Alkalis	0.2500%
Chlorides (Cl)	0.0050%
Sulphates (SO_4)	0.0500%
Aluminum (Al)	0.0250%
Calcium (Ca)	0.0050%

Methods of Testing

Alkalis.—Dissolve 2 gm. of ammonium chromate in 100 mls of water. Add 50 mls of 10% solution of lead acetate, filter, remove the lead from the filtrate by passing in hydrogen sulphide gas and filter. Evaporate the lead-free filtrate to dryness and ignite and weigh the residue. The weight should not exceed 0.005 gm. The lead acetate used should be free from alkalis or a correction applied to the results for any it may contain.

Chlorides.—Dissolve 1 gm. in 20 mls of water, add 10 mls of nitric acid, warm the mixture to about 50°C ., add a few drops of silver nitrate solution and allow it to stand five minutes. No turbidity should be produced.

Sulphates.—Dissolve 2.5 gm. in 100 mls of water, add 25 mls of hydrochloric acid and some barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Aluminum.—Dissolve 2 gm. in 35 mls of water, add 5 mls of ammonia water, heat the mixture on a steam-bath until the odor of ammonia has almost disappeared and then allow it to stand at ordinary temperature for twelve to eighteen hours. No precipitate should form.

Calcium.—Dissolve 2 gm. in 30 mls of water, add 5 mls of ammonia water and some ammonium oxalate solution and allow it to stand two hours. No precipitate should form.

Quantitative Method.—Dissolve 0.1 to 0.2 gm. of ammonium chromate, accurately weighed, in water, add 2 gm. of potassium iodide and 10 mls of diluted sulphuric acid, dilute to about 400 mls with water and titrate the liberated iodine with tenth-normal sodium thiosulphate solution using starch solution as indicator. At least 99.5% of $(\text{NH}_4)_2\text{CrO}_4$ should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.0050695$ gm. of $(\text{NH}_4)_2\text{CrO}_4$, log 70497.

AMMONIUM FLUORIDE

NH_4F

Mol. Wt. 37.04

Ammonium fluoride occurs usually as colorless crystals, easily soluble in water but with difficulty in alcohol. It volatilizes readily on heating. In dry air ammonium fluoride is stable, but in the presence of water and moist air it gradually changes to the bifluoride with liberation of ammonia. Small amounts of bifluoride are nearly always present in the salt, imparting to its solutions an acid reaction.

Ammonium fluoride is principally used in the analysis of silicates, as a blow-pipe reagent and in certain fermentative processes to suppress undesirable secondary fermentations. It should be stored in well-stoppered bottles, which, if of glass, should have an interior coating of paraffin or other substance not susceptible to the action of the salt.

Maximum Limits of Impurities

Non-volatile Matter	0.0050%
Chlorides (Cl)	0.0025%
Sulphates (SO_4)	0.0025%
Silicofluorides (SiO_2)	0.4000%
Heavy Metals	Trace
Ammonium Bifluoride ($\text{NH}_4\text{F.HF}$)	5.0000%

Methods of Testing

Ammonium fluoride readily attacks glass with formation of silicofluoride. The tests given below should therefore be made as far as possible with the aid of platinum or other non-corrodable dishes.

Non-volatile Matter.—Ignite 10 gm. of the salt and weigh the residue. Not more than 0.0005 gm. should be found.

Chlorides.—Dissolve 2 gm. of ammonium fluoride in 20 mls of water and add a few drops of nitric acid and of silver nitrate solution. Not more than a slight opalescence should be produced.

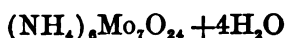
Sulphates.—Treat 10 gm. of the salt with 20 to 25 mls of hydrochloric acid and evaporate the mixture to dryness on the steam-bath. Take up the residue with 100 mls of water, add 5 mls of hydrochloric acid and some barium chloride solution and allow the mixture to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Silicofluorides.—Dissolve 5 gm. in 25 mls of water, add 2 mls of saturated solution of potassium chloride and then 35 mls of alcohol. Not more than a slight opalescence should be produced.

Heavy Metals.—Dissolve 4 gm. of the salt in 50 mls of water and divide the solution into two equal portions. To one portion add a few drops of hydrochloric acid and some hydrogen sulphide water. No change should be apparent. To the second portion add ammonia water to alkalinity and then a few drops of ammonium sulphide solution. No precipitate and not more than a slight greenish coloration should be produced.

Ammonium Bifluoride.—Titrate a weighed sample of the ammonium fluoride with standard alkali solution, using phenolphthalein as indicator. Not over 5% of $\text{NH}_4\text{F} \cdot \text{HF}$ should be found.

1 mil of normal $\text{NaOH} = 0.05705$ gm. of $\text{NH}_4\text{F} \cdot \text{HF}$,
log 75626.

AMMONIUM MOLYBDATE

Mol. Wt. 1236.32

Ammonium molybdate usually occurs as colorless or slightly greenish or yellowish crystals, easily soluble in water. It contains about 81% of MoO_3 .

When gently ignited the salt loses water and ammonia and a residue of molybdic anhydride, MoO_3 , is left. This serves as an easy method of determining approximately the percentage of MoO_3 in the salt.

Ammonium molybdate is used chiefly in the detection and determination of phosphates, arsenates, lead, and bismuth; as a reagent for alkaloids, albumin, alcohol, hydrogen peroxide, tannin, uric acid, stannous chloride, free mineral acids and many other substances; for detecting saccharose in lactose, and nickel in the presence of a large amount of cobalt.

Maximum Limits of Impurities

Phosphates (P_2O_5)	0.0005%
Sulphates (SO_3)	0.0500%
Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0032%
Heavy Metals	0.0000%

Methods of Testing

Phosphates.—Dissolve 10 gm. in a mixture of 20 mls of ammonia water and 25 mls of water, add this solution slowly and with constant stirring to 150 mls of nitric acid and allow the mixture to stand twelve to eighteen hours at about 40°C . No yellow precipitate should form.

Sulphates.—Dissolve 10 gm. in 25 mls of ammonia water, pour the solution gradually and with stirring into 60 mls of nitric acid, add barium nitrate solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Chlorides.—Dissolve 2 gm. in 10 mls of water, add the solution gradually to 10 mls of nitric acid and to the mixture add silver nitrate solution. The liquid should not become more than opalescent.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add a few milligrams of sodium chloride and one drop of indigo solution and mix the solution with 10 mls of sulphuric acid. The blue color should persist after mixing. ●

Heavy Metals.—Dissolve 1 gm. in 5 mls of water, add 5 mls of ammonia water and a few drops of ammonium sulphide solution. No precipitate or green color should be produced.

Quantitative Method.—Determine the molybdic anhydride by the method described under Acid Molybdic, 85%. At least 81% of MoO_3 should be found.

AMMONIUM MOLYBDATE SOLUTION

Ammonium molybdate solution is a clear, colorless or slightly yellowish liquid having a strongly acid reaction and a slight odor of nitric acid. It may be prepared as follows: Dissolve 100 gm. of molybdic acid, 85%, in a mixture of 144 mls of 28% ammonia water and 271 mls of water and pour this solution slowly and with constant stirring into a mixture of 489 mls of nitric acid, sp. gr. 1.42, and 1148 mls of water. Keep the mixture in a warm place for several days or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment which may have deposited.*

Ammonium molybdate solution finds its principal use in the detection and determination of phosphorus in the form of orthophosphate. Metaphosphates and pyrophosphates do not react with it until converted to orthophosphates by the nitric acid of the solution.

The reagent should be stored in glass-stoppered bottles, the stoppers of which are lubricated with a little petrolatum to keep them from sticking. On keeping, the solution oc-

* This is the formula adopted by the Association of Official Agricultural Chemists and published in Bull. No. 107 (Revised) of the Dept. of Agriculture, Bureau of Chemistry (1912). Other formulas are often employed and are described by various authors.

asionally is observed to deposit white to yellowish crystalline material which forms crusts on the bottom or sides of the container. This material has been identified as hydrated molybdic acid, $\text{H}_2\text{MoO}_4 + \text{H}_2\text{O}$, and as its quantity increases, the solution, thus gradually deprived of its essential constituent, loses its efficiency. A solution showing a considerable deposit should be rejected.

AMMONIUM NITRATE

NH_4NO_3

Mol. Wt. 80.05

Ammonium nitrate is usually in the form of colorless crystals or a white granular powder, soluble in less than 1 part of water and in about 25 parts of alcohol.

The salt is used principally as an oxidizing agent, especially in the combustion of organic matter, and in coke analysis. It is also employed in phosphorus determination by the molybdate method and, in the molten state, as a solvent for metals.

Ammonium nitrate should be stored in well-stoppered bottles as it is somewhat deliquescent.

Maximum Limits of Impurities

Non-volatile Matter	0.0100%
Phosphates (P_2O_5)	0.0010%
Arsenates (As_2O_5)	0.0050%
Sulphates (SO_3)	0.0050%
Sulphocyanates (CNS)	0.0250%
Tar Bases	0.0000%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0005%
Nitrites (N_2O_2)	0.0005%

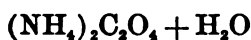
Methods of Testing

Non-volatile Matter, Phosphates, Arsenates, Sulphates, Sulphocyanates, Tar Bases and Heavy Metals.—Apply the tests prescribed under the methods of testing Ammonium Chloride. The results should be as there stated. Test the ammonium nitrate further as follows:

Chlorides.—Dissolve 2 gm. in 20 mls of water, add a few drops of nitric acid and some silver nitrate solution. The mixture should be clear.

Nitrates.—Dissolve 1 gm. in 20 mls of water, add 1 mil of diluted sulphuric acid and 1 mil of metaphenylenediamine solution. A yellow or yellowish-brown color should not develop.

AMMONIUM OXALATE



Mol. Wt. 142.10

Ammonium oxalate is usually in the form of colorless crystals, soluble in about 25 parts of water.

The salt is used principally in the detection and determination of calcium and rare metals (cerium, thorium, zirconium, etc.); in the separation of calcium and strontium; to precipitate lead, zinc, barium and calcium and to separate them from vanadic acid; and in the estimation of quinine.

Maximum Limits of Impurities

Non-volatile Matter	0.0333%
Sulphates (SO_4)	0.0050%
Chlorides (Cl)	0.0050%
Heavy Metals	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 3 gm. of ammonium oxalate. Not more than 0.001 gm. of residue should remain.

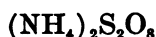
Sulphates.—Dissolve 5 gm. in 200 mls of water, heat the solution to boiling, add 10 mls of hydrochloric acid and some barium chloride solution and allow the mixture to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Chlorides.—Dissolve 1 gm. in 25 mls of water and add 10 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should develop.

Heavy Metals.—Dissolve 1 gm. in 25 mls of water and add 10 mls of hydrogen sulphide water. No change in

color should be produced. Then add 5 mls of ammonia water. No green color should result and the liquid should remain clear.

AMMONIUM PERSULPHATE



Mol. Wt. 228.19

Ammonium persulphate is usually in the form of colorless crystals or a white granular powder, very easily soluble in water (about 2 parts). It contains at least 95% of $(\text{NH}_4)_2\text{S}_2\text{O}_8$.

Ammonium persulphate in perfectly dry condition is stable but exposed to moist air or to heat it decomposes with liberation of oxygen, at times with violence. Contact with organic substances, as alcohol, also effects similar decomposition. In aqueous solution it decomposes slowly when cold, quickly when warm. A bluish-violet fluorescence in the aqueous solution indicates impurity of the salt.

Ammonium persulphate is a useful oxidizing agent in analysis and for removal of organic matter. Among its various uses may be mentioned the separation of chromium and manganese and their determination in steel; the separation of iron, chromium and aluminum in mixtures of their hydroxides; the separation of manganese from nickel, copper and cadmium; of cobalt from nickel; of chlorine, bromine and iodine; and the determination of lead.

Ammonium persulphate should be kept dry by storage in tightly-stoppered bottles in a cool place.

Maximum Limits of Impurities

Non-volatile Matter	0.1000%
Chlorides (Cl)	0.0010%
Manganese (Mn)	0.0002%
Other Heavy Metals	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of ammonium persulphate. Not over 0.002 gm. of residue should remain.

Chlorides.—Dissolve 5 gm. in 20 mls of water and add a few drops of silver nitrate solution, avoiding an excess. Not more than a slight opalescence should be produced in the mixture.

Manganese.—Mix 1.5 mls of silver nitrate solution with 20 mls of water and 1 mil of nitric acid, heat the mixture to 80° to 90° C., add 5 gm. of ammonium persulphate, continue heating at the same temperature for about one minute and then cool the mixture quickly. No red color should develop.

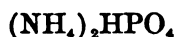
Other Heavy Metals.—Dissolve 2 gm. of ammonium persulphate in 25 mls of sulphurous acid, evaporate the solution to dryness on the steam-bath and dissolve the residue in 40 mls of water. To 20 mls of the solution add hydrogen sulphide water. No color or precipitate should be produced.

To the remaining 20 mls of the solution prepared as directed in the preceding paragraph, add ammonia water in excess and a few drops of ammonium sulphide solution. No precipitate should form at once although a green color may be visible.

Sulphurous acid may at times contain traces of iron which react in this test. When sulphurous acid, free from iron, is not available, a solution of 1 gm. of sodium bisulphite in 25 mls of water may be substituted for the acid in making the test.

Quantitative Method.—Dissolve about 0.3 gm. of the salt, accurately weighed, in 25 mls of 10% potassium iodide solution, add 10 mls of diluted sulphuric acid, allow the mixture to stand half an hour and titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 95% of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.011411$ gm. of $(\text{NH}_4)_2\text{S}_2\text{O}_8$, log 05729

AMMONIUM PHOSPHATE**(Secondary Ammonium Phosphate)**

Mol. Wt. 132.13

Ammonium phosphate is in the form of colorless crystals or a white crystalline powder, soluble in about 4 parts of water. It contains a least 95% of $(\text{NH}_4)_2\text{HPO}_4$. The salt usually contains some of the primary phosphate, $\text{NH}_4\text{H}_2\text{PO}_4$, due to the loss of ammonia which gradually takes place when the secondary ammonium phosphate is exposed to the air.

Ammonium phosphate is used principally as a precipitant for magnesium, nickel, zinc and uranium. It should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Alkalis	0.1500%
Arsenic (As)	0.0015%
Sulphates (SO_4)	0.0050%
Chlorides (Cl)	0.0020%
Nitrates (N_2O_5)	0.0016%
Heavy Metals	0.0000%

Methods of Testing

Alkalis.—Dissolve 2 gm. in 100 mls of water, precipitate the phosphoric acid with a slight excess of lead acetate solution, filter, remove the lead from the filtrate with hydrogen sulphide gas, again filter, evaporate the filtrate and ignite and weigh the residue. Not more than 0.003 gm. should be found.

Arsenic.—Shake 1 gm. of the powdered ammonium phosphate with 3 mls of stannous chloride solution and let the mixture stand one hour. No dark color should develop.

Sulphates.—Dissolve 5 gm. in 100 mls of water, add 5 mls of hydrochloric acid and a few mls of barium chloride solution and allow the mixture to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Chlorides.—Dissolve 5 gm. in 25 mls of water, add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should develop.

Nitrates.—Dissolve 2 gm. in 10 mls of water, add a granule of sodium chloride, 1 drop of indigo solution and 10 mls of sulphuric acid. The blue color should persist after mixing.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water, acidify with hydrochloric acid and add hydrogen sulphide water. The mixture should be clear and colorless.

Dissolve 2 gm. in 20 mls of water and add ammonium sulphide solution. No precipitate or dark color should result.

Quantitative Determination.—Titrate a solution of the salt with normal hydrochloric acid solution, using methyl orange as indicator. At least 95% of $(\text{NH}_4)_2\text{HPO}_4$ should be found.

1 mil of normal $\text{HCl} = 0.13213$ gm. of $(\text{NH}_4)_2\text{HPO}_4$,
log 12100.

AMMONIUM SULPHATE

$(\text{NH}_4)_2\text{SO}_4$

Mol. Wt. 132.14

Ammonium sulphate occurs as colorless crystals or white granules, soluble in about 2 parts of water but insoluble in alcohol.

It is used largely as a precipitant of albuminoids and urates; in connection with ammonium oxalate for separating calcium and strontium; for transposing organic acids in the analysis of their salts; and as a standard in nitrogen determinations.

Maximum Limits of Impurities

Non-volatile Matter	0.0100%
Chlorides (Cl)	0.0005%
Nitrates (N_2O_5)	0.0016%
Phosphates (P_2O_5)	0.0010%

Arsenates (As_2O_3)	0.0050%
Sulphocyanates (CNS)	0.0250%
Heavy Metals	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 5 gm. of ammonium sulphate. Not more than 0.0005 gm. of residue should remain.

Chlorides.—Dissolve 2 gm. in 20 mls of water and add a few drops of nitric acid and silver nitrate solution. The liquid should remain clear.

Nitrates.—Dissolve 2 gm. in 10 mls of water add a few milligrams of sodium chloride, one drop of indigo solution and finally 10 mls of sulphuric acid. The mixture should retain a blue color.

Phosphates and Arsenates.—Dissolve 5 gm. in 20 mls of water, add 3 mls of magnesia mixture and 10 mls of ammonia water and allow to stand about fifteen hours. No precipitate should form.

Sulphocyanates.—Dissolve 5 gm. in 20 mls of water and add 2 mls of hydrochloric acid and a drop of ferric chloride solution. The liquid should not become red.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water, add a few drops of hydrochloric acid and 10 mls of hydrogen sulphide water. No coloration should take place. Add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

AMMONIUM SULPHIDE SOLUTION

(Ammonium Sulphydrate Solution)

Ammonium sulphide solution is a colorless or yellowish liquid having a sulphidic and ammoniacal odor and a strongly alkaline reaction upon litmus paper. It contains at least 8% of sulphidic S.

The solution is made by passing hydrogen sulphide gas into ammonia water and consists generally of a mixture of sulphide and sulphhydrate of ammonium. The reagent when first prepared is colorless but on keeping, especially in contact with air, it becomes yellow, due to the formation of polysulphides. On long standing further decomposition takes place and sulphur is deposited. The ammonium polysulphide solution required for the separation of the sulphides of the tin and copper groups and other purposes, may be prepared by dissolving sulphur in this reagent.

Solution of ammonium sulphide is used chiefly as a group reagent and for the detection of the heavy metals; also in the detection of hydrocyanic acid.

The solution should be stored in a cool, dark place in well-stoppered bottles preferably of dark amber glass, and should be exposed to the action of the air as little as possible. Upon the appearance of a notable deposit of sulphur the solution should be rejected.

Maximum Limits of Impurities

Arsenic (As)	0.0060%
Antimony (Sb)	0.0007%
Tin (Sn)	0.0013%
Non-volatile Matter	0.0200%
Carbonates (CO ₂)	0.0050%
Chlorides (Cl)	0.0050%

Methods of Testing

Arsenic, Antimony and Tin.—Add an excess of hydrochloric acid to 50 mls of the ammonium sulphide solution. Hydrogen sulphide gas is evolved but no colored precipitate should form.

Non-volatile Matter.—Evaporate 10 mls of the solution and ignite and weigh the residue. Not more than 0.002 gm. should be found.

Carbonates.—To 10 mls of the solution add 3 mls of calcium chloride solution and warm the mixture. No precipitate should form.

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Chlorides.—To 0.2 mil of the ammonium sulphide solution add 5 mils of ammonia water and 15 mils of tenth-normal silver nitrate solution, filter and acidulate the filtrate with nitric acid. Not more than an opalescence should be produced.

Quantitative Method.—Dilute a weighed portion (5 to 10 gm.) to 100 mils. Transfer an aliquot part of this dilution, representing about 0.5 gm. of the original ammonium sulphide solution, to a graduated flask of 100 mils capacity, add 20 mils of ammonia water and 50 mils of tenth-normal silver nitrate solution and dilute with water to the mark. Thoroughly mix and filter. To 50 mils of the filtrate add 20 mils of nitric acid and some solution of ferric ammonium sulphate and titrate with tenth-normal ammonium sulphocyanate solution. At least 8% of S should be found.

1 mil of tenth-normal AgNO_3 = 0.001603 gm. of S,
log 20493.

AMMONIUM SULPHOCYANATE

(Ammonium Thiocyanate)

NH_4CNS

Mol. Wt. 76.12

Ammonium sulphocyanate is usually in the form of colorless crystals, easily soluble in water (less than 1 part) and in alcohol. The salt is deliquescent. The solubility in alcohol serves to detect less soluble salts, such as the chloride, sulphate, etc., when present in any considerable amount.

Ammonium sulphocyanate is used principally as a reagent for iron and also in forensic and other analyses for the detection and determination of small quantities of arsenic, antimony, mercury, silver, copper, halogens and mustard oil.

The salt should be stored in tightly-stoppered containers and should not be exposed to dust which, through the presence of iron, may cause discoloration of the reagent.

Maximum Limits of Impurities

Non-volatile Matter	0.0250%
Substances Insoluble in Alcohol	0.0000%
Sulphates (SO ₃)	0.0100%
Chlorides (Cl)	0.0050%
Heavy Metals	0.0000%
Iron (Fe)	0.0005%

Methods of Testing

Non-volatile Matter.—Ignite 4 gm. of ammonium sulphocyanate. The residue should not weigh more than 0.001 gm.

Substances Insoluble in Alcohol.—Dissolve 1 gm. in 10 mls. of absolute alcohol. The solution should be clear and free from insoluble residue.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add a few drops of hydrochloric acid and some barium chloride solution. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 100 mls of water, add 25 mls of nitric acid, heat on the steam-bath for three or four hours until the sulphocyanate is decomposed and add silver nitrate solution. Not more than a slight opalescence should be produced.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Iron.—Dissolve 1 gm. in 20 mls of water and add a few drops of hydrochloric acid. The solution should remain colorless.

ANILINEC₆H₅NH₂

Mol. Wt. 93.10

Aniline for reagent purposes is a colorless, or very nearly colorless, oily liquid, having a specific gravity of about 1.027 (theory 1.0254 at 15°/15° C.) and boiling at 183° to 184° C. (theory 183.7° C.). It is soluble in about 35 parts of water. In a freezing mixture it solidifies, melting at about —8° C. (theory —5.96° C.).

The most common impurity in aniline is toluidine the presence of which in any considerable quantity affects the boiling point of the aniline, toluidine boiling at about 200° C.

Aniline is employed in a large number of qualitative reactions such as the detection of furfural, aldehyde, fusel oil, phenol, chloroform, hydrated chloral, pentoses, persulphates, wood fiber, etc. A standard solution is used in the volumetric estimation of nitrous acid. It is also used as a reagent in microscopy, in the preparation of staining solutions and for preparing aniline salts and derivatives.

Aniline is very sensitive to light and air and soon turns brown when exposed to their action; it should, therefore, be kept in well-stoppered containers with very little air space and should be protected from light.

Maximum Limits of Impurities

Hydrocarbons	0.0000%
Nitrobenzene	0.0000%

Method of Testing

Hydrocarbons and Nitrobenzene.—Dissolve 5 mls of aniline in 10 mls of hydrochloric acid. A clear solution should result. Then dilute with 15 mls of water and cool the mixture. The liquid should not become cloudy.

ARSENIC TRIOXIDE

(Arsenous Acid; (Acid) Arsenous Anhydride)

As₂O₃

Mol. Wt. 197.92

Arsenic trioxide usually occurs as a white powder, often as amorphous, transparent, glassy lumps, white, opaque crystalline pieces or a mixture of the two. It is usually soluble in about 50 to 60 parts of water, but the solubility varies considerably, the amorphous form being more soluble than the crystalline; the presence of hydrochloric acid increases the solubility in water. In alcohol or ether it is slightly soluble; readily in solutions of the alkali hydroxides

or carbonates with the formation of arsenite. The reagent contains at least 99.8% of As_2O_3 .

On heating, the glassy variety melts before it begins to sublime appreciably, while the crystalline form sublimes without previously melting. The sublimed arsenic trioxide condenses in octahedral crystals, the characteristic form of which permits the microscopical identification of traces of arsenic, such as those obtained in the reduction tube of the Marsh apparatus. Arsenic trioxide is not changed in the air, but its solution in alkali (arsenite) gradually oxidizes on exposure to the air, forming arsenate.

The reagent is used principally in preparing standard solutions for iodometric titrations and has been proposed as a starting material for alkalimetric solutions. It is also employed as a reducing agent and for the detection of acetic acid in the presence of other volatile organic acids. Regarding the preparation of arsenic trioxide for volumetric and other analytical purposes see Chapin, *Journal Industrial and Engineering Chemistry*, 10, 522 (1918), whose suggestions as to tests of purity are embodied in the methods of testing given below.

Arsenic trioxide should be stored and handled with due regard for its poisonous nature. Sublimation should be performed in a well-ventilated hood. Alkaline solutions should be prepared without excessive heat to avoid oxidation, and should be stored in well-stoppered bottles with as little air space as possible.

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Carbonaceous Matter	0.0000%
Insoluble Matter	0.0000%
Foreign Heavy Metals	0.0000%
Antimony Oxide (Sb_2O_3)	0.1500%
Sulphides (S)	0.0050%

Methods of Testing

Non-volatile and Carbonaceous Matter.—Heat 1 gm. cautiously in a porcelain dish under a hood to slight redness

and weigh any non-volatile residue. The weight should not exceed 0.0005 gm. During the sublimation no darkening of the arsenic trioxide should be apparent.

Insoluble Matter.—Powder the arsenic trioxide if it is not already a powder, introduce 1 gm. into a test-tube which has been previously moistened with water to prevent the substance caking on the glass, add 10 mls of ammonia water and heat to very gentle boiling, shaking the tube meanwhile. A perfectly clear and colorless solution should result.

Foreign Heavy Metals.—To the solution obtained in the preceding test add 10 mls of hydrogen sulphide water, mix and heat just to boiling. No precipitate and not more than a faint yellow color should be produced.

Antimony Oxide.—Cool the solution obtained in the preceding test at once by immersing the tube in water, and when cold place it in ice water and allow it to stand fifteen minutes. No turbidity should be apparent in the solution at the end of this time.

Sulphides.—Dissolve 1 gm. in 10 mls of normal sodium hydroxide solution, add 1 drop of lead acetate solution and mix. No coloration should be produced.

Quantitative Method.—Dry the arsenic trioxide to constant weight at 100° C., weigh out accurately about 0.2 gm. of the dried material, dissolve it in 20 mls of boiling water by the gradual addition of sodium hydroxide solution until complete solution results. Neutralize the solution with dilute sulphuric acid, using phenolphthalein as indicator, cool, dissolve in it 2 gm. of sodium bicarbonate and titrate the mixture with tenth-normal iodine solution, using starch solution as indicator. At least 99.8% of As_2O_3 should be found.

1 mil of tenth-normal I = 0.004948 gm. of As_2O_3 ,
log 69443.

AZOLITMIN

Azolitmin is a water-soluble coloring matter obtained from litmus and usually occurs as dark violet scales. It is soluble with some difficulty in water; insoluble in alcohol or dilute acids, but very soluble in dilute alkalis forming deep blue solutions.

Azolitmin is the coloring matter of litmus upon which depends the value of the latter as an indicator. Azolitmin has the advantage over litmus of being a pure principle unmixed with other coloring matters which would detract from its sensitiveness, and therefore is very convenient for the preparation of an efficient test solution. A 1% solution is usually employed and may be made by dissolving 1 gm. of azolitmin in 80 mls of hot water and then adding 20 mls of alcohol.

Azolitmin is of service in the volumetric determination of alkalis and alkaline earths in the absence of carbonates; of most mineral acids and some organic acids (not tartaric or citric acid); and of the salts of some alkaloids. It is also used in the preparation of litmus media for bacteriological purposes.

The solution is best kept in partly filled bottles stoppered with a pledget of cotton to allow access of air. When air is excluded, the solution soon deteriorates and loses its characteristic coloring properties; when it is thus changed it may sometimes be restored to good condition by free exposure to the air.

Method of Testing

Add 0.1 mil of the azolitmin solution, prepared as above described, to 50 mls of water, free from carbon dioxide. The bluish-red color of the liquid should be changed to red by the addition of 0.05 mil of tenth-normal hydrochloric acid solution, and should be changed to bluish violet by the addition of 0.05 mil of tenth-normal sodium hydroxide solution.

BARIUM ACETATE

Mol. Wt. 273.45

Barium acetate is usually in the form of a white, crystalline powder. It is soluble in about 2 parts of water, slightly soluble in alcohol.

Barium acetate is employed chiefly as a precipitant for sulphates and chromates. It is especially adapted to those cases in which it is desired to transpose sulphates to acetates for special treatment, as in separating alkalis from magnesium.

The salt should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0032%
Calcium and Alkalis	0.1000%
Heavy Metals	0.0000%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add a few milligrams of sodium chloride and a drop of indigo solution and mix the liquid with 10 mls of sulphuric acid. The blue color should persist after mixing.

Calcium and Alkalis.—Dissolve 5 gm. of barium acetate in 200 mls of water, add 2 mls of hydrochloric acid, heat to boiling, add 25 mls of diluted sulphuric acid and allow to stand about eighteen hours. Filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.005 gm.

Heavy Metals.—Dissolve 2 gm. in 40 mls of water. To half the solution add hydrogen sulphide solution and to the other half a few drops each of ammonia water and of ammonium sulphide solution. No dark color or precipitate should be produced in either portion.

BARIUM CARBONATE**BaCO₃****Mol. Wt. 197.37**

Barium carbonate is usually a white powder, almost insoluble in water free from carbon dioxide; slightly soluble (about 1:1000) in carbonated water. In hydrochloric, nitric or acetic acid it dissolves with effervescence. The reagent contains at least 99.7% of BaCO₃.

Barium carbonate is used principally in the separation of iron and aluminum from manganese, zinc, magnesium and calcium; for removing sulphuric acid from solutions; and for preparing other barium salts.

Maximum Limits of Impurities

Acid-insoluble Substances	0.0000%
Barium Hydroxide	0.0000%
Calcium and Alkalis	0.1000%
Iron (Fe)	0.0040%
Other Heavy Metals	0.0000%
Chlorides (Cl)	0.0010%
Nitrates (N ₂ O ₅)	0.0032%

Methods of Testing

Acid-insoluble Matter (Barium Sulphate).—Dissolve 5 gm. in 100 mls of 10% hydrochloric acid. The solution should be clear and colorless, or very nearly colorless.

Barium Hydroxide.—Shake 1 gm. repeatedly during half an hour with 20 mls of water (free from carbon dioxide), filter and test the filtrate with litmus paper. It should not show an alkaline reaction.

Calcium and Alkalis.—Dissolve 5 gm. in a mixture of 10 mls of hydrochloric acid and 200 mls of water, heat the solution to boiling, add 25 mls of 10% sulphuric acid, allow to stand twelve to eighteen hours, filter, evaporate the filtrate and ignite and weigh any residue obtained. The weight should not exceed 0.005 gm.

Iron.—Dissolve 0.5 gm. in 20 mls of water with the aid of 3 to 4 mls of hydrochloric acid and add potassium

sulphocyanate solution. Not more than a slight red color should result.

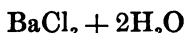
Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water with the aid of 3 to 4 mls of hydrochloric acid and add hydrogen sulphide water. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 20 mls of water with the aid of 5 mls of nitric acid and add silver nitrate solution. No change should be apparent.

Nitrates.—Dissolve 1 gm. in 10 mls of 36% acetic acid, add 1 drop of indigo solution, a few milligrams of sodium chloride and 10 mls of sulphuric acid and shake the mixture. A blue color should persist after the mixing.

Quantitative Method.—Dissolve the barium carbonate in an excess of standard acid solution and titrate the excess acid with standard alkali solution, using methyl orange as indicator. At least 99.7% of BaCO_3 should be found. 1 mil of normal HCl = 0.098685 gm. of BaCO_3 , log 99425.

BARIUM CHLORIDE



Mol. Wt. 244.32

Barium chloride occurs usually as colorless crystals, soluble in about 2.5 parts of water but insoluble in alcohol. The presence of hydrochloric acid decreases the solubility in water. Its aqueous solution is neutral to litmus.

The salt is ordinarily unchanged on exposure to the air, but in a stream of warm, dry air it is somewhat efflorescent and in air saturated with moisture it is deliquescent. At 100° to 120° C., it is rendered anhydrous.

Barium chloride is used principally in the detection and determination of sulphates, both by gravimetric and volumetric means. The presence of much hydrochloric acid considerably lessens the delicacy of the reagent in the detection of small amounts of sulphates. It is also employed in estimating lactic acid in wine; in the detection of fluorides

and chromates; and in the standardization of soap solutions used in water analysis.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Alkali Salts	0.1000%
Strontium and Calcium Chlorides	0.2000%
Heavy Metals	0.0000%
Nitrates (N_2O_5)	0.0032%
Chlorates (Cl_2O_5)	0.0500%

Methods of Testing

Alkali Salts.—Dissolve 2 gm. in about 100 mls of water, add 1 mil of hydrochloric acid, heat to boiling, add 10 mls of 10% sulphuric acid and allow it to stand at least 12 hours. Filter, evaporate the filtrate and ignite and weigh any residue remaining. The weight should not exceed 0.002 gm.

Strontium and Calcium Chlorides.—Powder 1 gm. of the barium chloride, shake it with 20 mls of absolute alcohol for five minutes, filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add 2 or 3 drops of hydrochloric acid and some hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add 1 drop of indigo solution and 10 mls of sulphuric acid and shake the mixture. The blue color should not disappear at once.

Chlorates.—Powder 1 gm. of the barium chloride and warm it with 10 mls of hydrochloric acid in a test-tube. No odor of chlorine should be perceptible and neither the acid nor the salt should acquire a yellow color.

BARIUM DIOXIDE**(Barium Peroxide; Barium Superoxide)****BaO₂****Mol. Wt. 169.37**

Barium dioxide is a white or grayish-white powder, insoluble in water but soluble in dilute acids with the formation of hydrogen peroxide. It contains at least 85% of BaO₂.

Barium dioxide yields oxygen when ignited or when kept in contact with water. Carbon dioxide transforms it to the carbonate with loss of oxygen. When rubbed with organic matter it explodes.

Barium dioxide is used principally for preparing oxygen and hydrogen peroxide; also as a bleaching agent and for the detection of iodine and indican in urine. It should be stored in air-tight and moisture-proof containers, and kept from contact with organic matter.

Method of Testing

Quantitative Method.—Mix about 0.3 gm. of the barium dioxide, accurately weighed, with 25 mls of 10% potassium iodide solution, add 5 mls of hydrochloric acid to the mixture and allow it to stand about half an hour, shaking frequently. Titrate the liberated iodine with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 85% of BaO₂ should be found.

1 mil of tenth-normal Na₂S₂O₃ = 0.0084685 gm. of BaO₂,
log 92780.

BARIUM HYDROXIDE**Ba(OH)₂ + 8H₂O****Mol. Wt. 315.51**

Barium hydroxide is usually in the form of colorless or white crystals, soluble in about 20 parts of water, nearly always leaving traces of barium carbonate undissolved. It should contain at least 99% of Ba(OH)₂ + 8H₂O. When exposed to the air barium hydroxide effloresces and the

crystals become opaque. It also readily takes up carbon dioxide from the air, becoming thereby gradually transformed to the carbonate.

Barium hydroxide is used principally as a precipitant for magnesium; in the fusion of silicates; for saponifying fats; and in the determination of alkalis and tryptophan. Its tenth-normal and other standard solutions are employed in volumetric analysis.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Calcium and Alkalis	0.2000%
Chlorides (Cl)	0.0050%
Sulphides (S)	0.0030%
Heavy Metals	0.0000%

Methods of Testing

Calcium and Alkalis.—Dissolve 2.5 gm. of barium hydroxide in 100 mls of water, add 5 mls of hydrochloric acid, heat to boiling and add a slight excess of diluted sulphuric acid. Allow the mixture to stand for at least 12 hours, filter, evaporate the filtrate and ignite and weigh the residue. Not more than 0.005 gm. should be found.

Chlorides.—Dissolve 1 gm. in 25 mls of water, acidulate with 5 mls of nitric acid and add silver nitrate solution. Not more than a slight opalescence should develop.

Sulphides.—Dissolve 1 gm. in 20 mls of water, acidulate with 2 mls of hydrochloric acid and add lead acetate solution. No dark color should be produced.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water, acidulate with 2 mls of hydrochloric acid and add 10 mls of hydrogen sulphide water. No dark color or precipitate should be produced. Then add ammonia water in slight excess. No dark color should develop.

Quantitative Method.—Dissolve the barium hydroxide in water and titrate the solution with standard hydrochloric

acid solution, using methyl orange as indicator. At least 99% of $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$ should be found.

1 mil of normal $\text{HCl} = 0.157755$ gm. of $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$,
log 19799.

BARIUM HYDROXIDE SOLUTION

(Baryta Water)

Barium hydroxide solution is a clear, colorless liquid of strongly alkaline reaction. It should contain at least 3.3% of $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$.

When exposed to the air, barium hydroxide solution readily takes up carbon dioxide and becomes cloudy, due to the formation of barium carbonate. The white scales sometimes seen on the sides of bottles in which the solution has been kept for some time have been found to consist of barium sulphate, the sulphate radical having been gradually abstracted from the glass.

Barium hydroxide solution is used mainly as an alkalinizing agent, for detecting carbon dioxide and in testing alcohol, chloroform and creosote. It should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Calcium and Alkalis	0.0067%
Chlorides (Cl)	0.0002%
Sulphides (S)	0.0001%
Heavy Metals	0.0000%

Methods of Testing

Calcium and Alkalis.—Acidulate 75 mls of the solution with 5 mls of hydrochloric acid, heat to boiling and add a slight excess of diluted sulphuric acid. Let it stand at least twelve hours, filter, evaporate the filtrate and ignite and weigh the residue; not more than 0.005 gm. should be found.

Chlorides.—To 30 mls of the solution add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should develop.

Sulphides.—Acidulate 30 mls with 2 mls of hydrochloric acid and add lead acetate solution. No dark color should be produced.

Heavy Metals.—Acidulate 30 mls with 2 mls of hydrochloric acid and add 10 mls of hydrogen sulphide water. No dark color or precipitate should be produced. Then add ammonia water in slight excess. No dark color should develop.

Quantitative Method.—Titrate the solution with standard hydrochloric acid solution, using methyl orange as indicator. At least 3.3% of $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$ should be found.

1 mil of normal $\text{HCl} = 0.157755$ gm. of $\text{Ba}(\text{OH})_2 + 8\text{H}_2\text{O}$,
log 19799.

BARIUM NITRATE

$\text{Ba}(\text{NO}_3)_2$

Mol. Wt. 261.39

Barium nitrate is usually obtained as colorless crystals, soluble in about 12 parts of water. The aqueous solution is neutral to litmus. The salt is slightly hygroscopic in moist air.

Barium nitrate is used chiefly as a precipitant for sulphates and as a standard for preparing soap solutions used in water analysis.

The salt should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0010%
Alkalis	0.0667%
Heavy Metals	0.0000%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add a few drops of nitric acid and some silver nitrate solution. No change should be apparent.

Alkalis.—Dissolve 3 gm. in 100 mls of water, add 2 mls of hydrochloric acid, heat to boiling, add 15 mls of

diluted sulphuric acid, allow to stand twelve to eighteen hours, filter, evaporate the filtrate in a porcelain dish and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No precipitate or dark color should be produced.

Dissolve 1 gm. in 20 mls of water and add a few drops of ammonia water and of ammonium sulphide solution. No dark coloration or precipitate should result.

BARIUM SULPHIDE

BaS

Mol. Wt. 169.43

Barium sulphide for reagent use is usually prepared in the form of cubes or lumps of a size adapted to use in gas generators. It contains at least 50% of BaS, mixed with other salts of barium, alkalis, etc. The reagent is used with hydrochloric acid, free from arsenic, for the preparation of arsenic-free hydrogen sulphide, and on treatment with hydrochloric acid a uniform current of the gas should be generated.

Barium sulphide should be stored in well-stoppered bottles to prevent deterioration by oxidation.

Maximum Limit of Impurity

Arsenic 0.0001%

Methods of Testing

Arsenic.—Heat 100 mls of nitric acid (sp. gr. 1.3) in a porcelain dish to about 75° C. and add 10 gm. of finely powdered barium sulphide, gradually, in quantities of 0.2 to 0.3 gm. at a time. When all has been added heat to boiling, add 100 mls of 20% sulphuric acid, evaporate, first on the steam-bath and finally on a sand-bath, until vapors of sulphuric acid begin to be evolved. Cool, stir the cooled residue with 100 mls of water, introduce the suspension a little at a time into a Marsh apparatus and allow the action

to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

Quantitative Method.—Dissolve 0.73 gm. of copper sulphate ($\text{CuSO}_4 + 5\text{H}_2\text{O}$) in 50 mls of water, add to the solution 1 gm. of powdered barium sulphide and, by drops, while stirring, 10 mls of 36% acetic acid. Filter, boil the filtrate to expel hydrogen sulphide and add an excess of ammonia water. No blue color should be produced.

BENZENE

(Benzol)

C_6H_6

Mol. Wt. 78.08

Benzene is a clear, colorless liquid of characteristic odor. It congeals between $+4^\circ$ and $+6^\circ$ C. (theory $+5.4^\circ$ C.), has a specific gravity of about 0.884 (theory 0.8799 at $20^\circ/4^\circ$ C.) and boils at about 80° C. (theory 80.2° C.). It is insoluble in water but soluble in alcohol and ether.

Benzene is used principally as a solvent for fats, resins, alkaloids, etc.; in the official method for analysis of sheep dips; and in forensic analysis. It should be stored in well-stoppered containers in a cool place, remote from fire.

Maximum Limits of Impurities

Thiophene	0.0000%
Carbon Disulphide (CS_2)	0.0075%

Methods of Testing

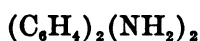
Thiophene.—Shake 50 mls of benzene with 20 mls of sulphuric acid. The acid should not become colored. Now add a few milligrams of isatin, shake thoroughly and allow to stand one hour. No green or blue color should be visible in the sulphuric acid.

Carbon Disulphide.—Thoroughly mix 50 mls of benzene with a solution of 5 gm. of potassium hydroxide in 50 mls of absolute alcohol and allow the mixture to stand several hours at about 20° C. Add about 100 mls of water,

shake the mixture, and after separation of the liquids, draw off the aqueous portion, neutralize it with acetic acid and add some copper sulphate solution. No precipitate should form.

BENZIDINE

(Para-diamino-diphenyl)



Mol. Wt. 184.17

Benzidine is a white or slightly reddish, crystalline powder. It is very difficultly soluble in cold water (about 2,500 parts), but more readily in boiling water, in alcohol and in ether (about 40 parts). It melts at about 128° C. (theory 127.5–8° C.). Benzidine is rather easily discolored on exposure to air and light which accounts for the slight reddish color often observed in the article.

The reagent is employed principally in the detection of blood; for the micro-chemical detection of erythrocytes and leucocytes; as a precipitant for sulphates, particularly in water analysis, and for the detection of lignin, wood-pulp, perchlorates, periodates, persulphates, percarbonates and perborates. Regarding the benzidine test as well as other tests for blood see Allen's Commercial Organic Analysis, 8, 524 (1913).

Benzidine should be stored in well-stoppered bottles of amber-colored glass.

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Sulphates (SO_4)	0.0125%

Methods of Testing

Non-volatile Matter.—Ignite 1 gm. of benzidine. Any residue remaining should not weigh more than 0.0005 gm.

Sulphates.—Dissolve 1 gm. in 50 mls of water, acidulated with 5 mls of hydrochloric acid, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Suitability for Detecting Blood.—Dissolve 1 gm. of benzidine in 10 mls of glacial acetic acid and to 1 mil of this solution add 10 mls of 3% hydrogen peroxide solution. No color, or at most a very faint color should appear within a few minutes. Then add a small amount of blood to the solution. A blue color should be produced.

BENZIN

(Petroleum Benzin; Petroleum Ether)

Benzin is a clear, colorless, non-fluorescent liquid of characteristic, petroleum-like odor. It has a specific gravity of 0.640 to 0.675 and distils completely between 35° and 80° C. It is highly inflammable and its vapor when mixed with air explodes if ignited.

Benzin is used largely as a solvent for fats, resins, alkalis, etc., and, on account of its immiscibility with water, for detecting that substance in various organic liquids. It should be stored in well-stoppered containers in a cool place, remote from fire.

Maximum Limits of Impurities

Non-volatile Matter	0.0030%
Acids	0.0000%
Sulphur Compounds and Reducing Substances	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 50 mls of benzin at about 50° C. Any residue remaining should not weigh over 0.001 gm.

Acids.—Shake 10 mls with 5 mls of water. The latter should not acquire an acid reaction to litmus paper.

Sulphur Compounds and Reducing Substances.—Mix 5 mls of benzin with 10 mls of alcoholic ammoniacal silver nitrate solution (see page ix) and heat to boiling for 5 minutes on the water bath, protected from the light. The liquid should not become brown.

BISMUTH SUBNITRATE

Bismuth subnitrate is a white powder consisting of basic bismuth nitrate of varying chemical composition and containing not less than 79%, and usually not more than 82% of Bi_2O_3 . It has an acid reaction to litmus paper and is insoluble in water but readily soluble in hydrochloric or nitric acid.

Bismuth subnitrate is principally used as a constituent of various reagent solutions for detecting albumin, sugars, alkaloids and potassium; it is also employed for converting arsenic sulphides into their corresponding acids.

The reagent should be stored in well-stoppered bottles as it is somewhat hygroscopic.

Maximum Limits of Impurities

Carbonates (CO_2)	0.0000%
Substances Insoluble in Sulphuric Acid	0.0000%
Copper (Cu)	0.0100%
Alkalis	0.2500%
Chlorides (Cl)	0.0100%
Sulphates (SO_4)	0.0175%
Ammonium Salts (NH_4)	0.0035%
Arsenic (As)	0.0010%

Methods of Testing

Carbonates and Substances Insoluble in Sulphuric Acid (Lead, etc.).—Dissolve 0.5 gm. in 25 mls of cold, diluted sulphuric acid. No evolution of carbon dioxide should be visible and the solution should be clear and free from insoluble matter.

Copper.—To 10 mls of the solution obtained in the preceding test add an excess of ammonia water and filter. The filtrate should be colorless.

Alkalis.—Dilute 10 mls of the solution obtained in the test for carbonates to about 100 mls and completely precipitate the bismuth by means of hydrogen sulphide gas. Filter, evaporate the filtrate to dryness and ignite and weigh the residue. It should not weigh more than 0.0005 gm.

Chlorides.—Dissolve 0.5 gm. of bismuth subnitrate in 5 mls of nitric acid and add a few drops of silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 2 gm. of bismuth subnitrate in 5 mls of nitric acid, dilute to 20 mls, add 0.5 mil of barium nitrate solution and allow the mixture to stand about fifteen hours. No precipitate of barium sulphate should form.

Ammonium Salts.—Heat to boiling a mixture of 1 gm. of bismuth subnitrate and 10 mls of 27% solution of sodium hydroxide. No vapors alkaline to litmus paper should be evolved.

Arsenic.—Ignite 1 gm. of bismuth subnitrate and dissolve the bismuth oxide obtained in sulphuric acid. Introduce the solution into a Marsh apparatus and allow the action to proceed for one-half hour. No deposit of arsenic should be visible at the end of that time.

Quantitative Method.—Dry the bismuth subnitrate over sulphuric acid, weigh out a portion of the dried material, ignite it and weigh the residue as Bi_2O_3 . At least 79% should be found.

BROMINE

Br

At. Wt. 79.92

Bromine is a dark reddish-brown, fuming liquid having a specific gravity of about 3.14 (theory 3.1883 at 0° C.), and, when perfectly pure, congealing at -7.3°C . and boiling at 58.7°C . These constants are affected to some extent by other halogens, small amounts of which are nearly always present. Bromine contains at least 98% of Br and not more than 2% of Cl.

Bromine is soluble in about 30 parts of water and is readily soluble in alcohol, ether, chloroform, or carbon disulphide. Its solubility in water is increased by the presence of alkali bromides. It dissolves in ammonia water with the formation of ammonium bromide and in alkali

hydroxide solutions, forming alkali hypobromite and bromide. Bromine volatilizes readily in the air at ordinary temperature, evolving highly irritating, reddish-brown vapors.

Bromine is used chiefly as an oxidizing agent; in the quantitative determination of manganese, sulphur, lead, nitrogen, resorcinol, urea, and formic and cinnamic acids; as a solvent for gold; and as a reagent for alkaloids, digitalin, phenol, uric acid and bile pigments.

The reagent should be stored in well-closed, glass-stoppered bottles in a cool place. The bottles should be placed in a second, larger container and the space between the bottle and the walls of the larger container filled with some incombustible mineral substance capable of absorbing the bromine or its vapor in case of breakage or leakage. Lead-lined boxes are often used for the storage of bottled bromine. Bromine quickly destroys wood, cork and other organic material and exerts an extremely caustic action upon the skin. It should therefore be stored and handled with the greatest care.

Maximum Limits of Impurities

Residue upon Evaporation	0.0100%
Sulphuric Acid (H_2SO_4)	0.0060%
Organic Bromine Compounds	0.0000%
Iodine (I)	0.1000%
Chlorine (Cl)	2.0000%

Methods of Testing

Residue upon Evaporation.—Volatilize 10 gm. in a porcelain dish on the steam-bath and weigh the residue. The weight should not exceed 0.001 gm.

Sulphuric Acid.—Dissolve 2 gm. in 30 mls of 5% sodium hydroxide solution, slightly acidulate the liquid with hydrochloric acid, boil it until all bromine vapors have been expelled and add barium chloride solution. No change should be apparent.

Organic Bromine Compounds.—Dissolve 2 gm. in 30 mls of 5% sodium hydroxide solution and allow it to stand

five or six hours. The solution should remain clear and no oily drops should separate from it.

Iodine.—Dissolve 1 gm. in 40 mls of water, add 4 gm. of powdered iron by hydrogen, shake two or three minutes, filter, to the filtrate add a few mls of starch solution and 0.5 to 1 mil of ferric chloride solution and allow it to stand ten minutes. No blue color should develop.

Quantitative Method and Determination of Chlorine Content.—Weigh a graduated 250 mil flask containing 15 gm. of potassium iodide and 30 mls of water, add about 1 mil of the bromine and weigh again. When the liberated iodine has completely dissolved, dilute the liquid to the mark, mix thoroughly and titrate an aliquot portion with tenth-normal sodium thiosulphate solution. At least 98% of Br and not more than 2% of Cl should be found.

Calculation: If A is the weight of the sample taken, I the total amount of iodine liberated and X the weight of the chlorine in A , then $X = \frac{I - 1.588 A}{1.9912}$ and $A - X =$ the weight of bromine in the sample.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.012692$ gm. of I,
log 10352.

BROMINE WATER

Bromine water is a yellowish-red, fuming liquid having a strong odor of bromine. It consists of a solution of bromine in water and contains at least 2.8 gm. of Br in each 100 mls. Exposed to the air it easily loses bromine by volatilization, especially when warmed. On keeping, particularly when exposed to sunlight, the bromine is gradually transformed into hydrobromic acid.

Bromine water is used chiefly as an oxidizing agent in analysis, for example in the detection of iodides and sulphites; in detecting phenol and alkaloids; and in the thalleioquin reaction for quinine.

The reagent should be stored in well-closed, glass-stop-

pered bottles, in a cool place, protected from light. A little undissolved bromine is often placed in the containers to keep the solution to full strength.

Maximum Limit of Impurity

Sulphuric Acid (H_2SO_4) 0.00002%

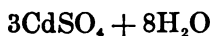
Methods of Testing

Sulphuric Acid.—Add 1 mil of hydrochloric acid and some barium chloride solution to 50 mils of bromine water, boil the liquid until the bromine has been completely expelled from it and allow it to stand two or three hours. No precipitate of barium sulphate should form.

Quantitative Method.—Mix about 10 mils, accurately measured, with a solution of 5 gm. of potassium iodide in 100 mils of water and allow the mixture to stand half an hour in a stoppered flask. Titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 0.5 gm. of Br per 100 mils of bromine water should be found.

1 mil of tenth-normal $Na_2S_2O_3 = 0.007992$ gm. of Br.,
log 90266.

CADMIUM SULPHATE



Mol. Wt. 769.51

Cadmium sulphate occurs usually as colorless, efflorescent crystals, soluble in less than 1 part of water and in about 2 parts of alcohol.

The reagent is used chiefly as a catalyst in the Marsh test for arsenic; in the determination of hydrogen sulphide; and for the detection of fumaric acid.

The salt should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Arsenic (As) 0.00013%
Foreign Heavy Metals 0.0000%

Methods of Testing

Arsenic.—Dissolve 8 gm. in 20 mls of water, introduce the solution into a Marsh apparatus and allow the action to proceed for half an hour. No deposit of arsenic should be visible in the reduction tube. The usual addition of cadmium sulphate in preparing the apparatus for the introduction of the chemical to be tested is omitted in this case.

Foreign Heavy Metals.—Dissolve 1 gm. in 20 mls of water and to one half the solution add an excess of ammonia water. A clear solution should result. Dilute the other half with 10 mls of water, add 5% sodium hydroxide solution to complete precipitation of the cadmium, filter and to the filtrate add hydrogen sulphide water. No change should be apparent. Acidulate the mixture slightly with hydrochloric acid. No color or precipitate should be produced.

CALCIUM CARBONATE

CaCO₃

Mol. Wt. 100.07

Calcium carbonate occurs as a white, crystalline powder. It is insoluble in water free from carbon dioxide, but is somewhat soluble in carbonated water, being reprecipitated on boiling. It dissolves readily in hydrochloric or nitric acid with effervescence. On ignition it is converted to calcium oxide.

Calcium carbonate is used chiefly in the detection and determination of chlorine, bromine, iodine and sulphuric acid in organic combination; in conjunction with ammonium chloride in decomposing silicates; in preparing calcium chloride solution for standardizing soap solutions used in water analysis; and as a catalyzer in the conversion of aliphatic acids to their corresponding ketones.

Maximum Limits of Impurities

Acid-insoluble Substances	0.0000%
Iron (Fe)	0.0040%
Other Heavy Metals	0.0000%

Magnesium (Mg)	0.0020%
Sulphates (SO_4)	0.0200%
Chlorides (Cl)	0.0010%
Phosphates (P_2O_5)	0.0010%
Alkalis and Calcium Oxide	0.0500%

Methods of Testing

Acid-insoluble Substances.—5 gm. of calcium carbonate should completely dissolve in 25 mls of hydrochloric or in 25 mls of nitric acid. The solutions should be clear and colorless or very nearly colorless.

Iron.—Dissolve 0.5 gm. in 20 mls of water with the aid of 3 to 4 mls of hydrochloric acid and add potassium sulphocyanate solution. Not more than a slight red color should develop.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water with the aid of 3 to 4 mls of hydrochloric acid and add hydrogen sulphide water. No change should be apparent.

Magnesium.—Dissolve 1 gm. in 5 mls of water with the aid of 3 to 4 mls of hydrochloric acid, add 10 mls of ammonia water and an excess of ammonium oxalate solution and allow it to stand four to five hours. Filter, to the filtrate add sodium phosphate solution and allow it to stand twelve to eighteen hours. No precipitate should form.

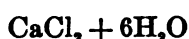
Sulphates.—Dissolve 1 gm. in a mixture of 5 mls of hydrochloric acid and 25 mls of water, boil the solution a few minutes, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 25 mls of water with the aid of 5 mls of nitric acid and add silver nitrate solution. No change should be apparent.

Phosphates.—Dissolve 10 gm. in 50 mls of nitric acid, add 25 mls of ammonium molybdate solution and allow it to stand twelve to eighteen hours at 30° to 40° C. No yellow precipitate should form.

Alkali Salts and Calcium Oxide.—Boil 4 gm. with 50 mls of water, filter, evaporate the filtrate and ignite and weigh any residue remaining. The weight should not exceed 0.002 gm.

CALCIUM CHLORIDE, CRYSTALS



Mol. Wt. 219.09

Calcium chloride, crystals, occurs as large, colorless, deliquescent crystals, soluble in less than 1 part of water; also soluble in alcohol. The 10% aqueous solution is neutral to litmus.

When heated to about 30° C. the salt melts, and on heating at higher temperatures it loses water of crystallization and is converted into a white, porous mass, which melts at about 800° C. When dissolved in water a marked reduction of temperature takes place; mixed with snow or ice a temperature of —45° C. may be reached.

The reagent is used chiefly in separating and detecting organic acids, detecting alcohol in certain esters and in volatile oils; and as a reagent for oxalic, malic and tartaric acids.

Calcium chloride, crystals, should be stored in tightly-stoppered bottles in a cool place.

Maximum Limits of Impurities

Substances Insoluble in Alcohol	0.0000%
Heavy Metals	0.0000%
Sulphates (SO ₄)	0.0050%
Nitrates (N ₂ O ₅)	0.0160%
Ammonium Salts (NH ₄)	0.0020%
Barium (Ba)	0.0020%
Arsenic (As)	0.0002%

Methods of Testing

Substances Insoluble in Alcohol.—Dissolve 2 gm. in 20 mls of alcohol. No insoluble residue should remain.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water and add a few drops of hydrochloric acid and some hydrogen

sulphide water. No change should be apparent. Then add 5 mls of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Sulphates.—Dissolve 2 gm. in 20 mls of water, and add a few drops of hydrochloric acid and some barium chloride solution. No precipitate should form.

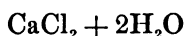
Nitrates.—Dissolve 1 gm. in 10 mls of water, add 0.2 mil of indigo solution and 10 mls of sulphuric acid and stir the mixture. A blue color should persist after stirring. The sulphuric acid should be added cautiously as hydrochloric acid gas is freely evolved.

Ammonium Salts.—Boil 2 gm. with 10 mls of 27% sodium hydroxide solution and test the vapors with moist red litmus paper. The paper should not turn blue.

Barium.—Dissolve 2 gm. in 20 mls of water, add 20 mls of calcium sulphate solution and allow it to stand two to three hours. No precipitate should form.

Arsenic.—Dissolve 5 gm. in 20 mls of water, introduce the solution into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should form in the reduction tube.

CALCIUM CHLORIDE, DRY, GRANULATED



Mol. Wt. 147.02

Calcium chloride, dry, granulated, occurs as white granular, porous masses about the size of peas. The salt is very hygroscopic and is used chiefly for drying gases. It should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Free Alkali (as CaO)	0.0028%
Arsenic (As)	0.0002%

Methods of Testing

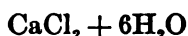
Free Alkali.—Dissolve 10 gm. in 100 mls of water and add a few drops of phenolphthalein solution. Should a

red color develop, add tenth-normal hydrochloric acid until it is discharged. Not more than 0.1 mil of the tenth-normal acid should be required.

Arsenic.—Perform the test as directed under Calcium Chloride, Crystals. The result should be as there indicated.

CALCIUM CHLORIDE, NEUTRAL

For Soil Analysis



Mol. Wt. 219.09

Neutral calcium chloride has the physical properties described under Calcium Chloride, Crystals.

The reagent is especially intended for use in conjunction with neutral zinc sulphide, for determining the acidity of soil according to the method of Truog, Bull. 249, Agri. Exp. Sta., Univ. Wisconsin (1915).

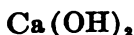
The salt should be stored in well-stoppered bottles in a cool place.

Method of Testing

Neutrality.—Dissolve 5 gm. in 100 mls of neutral water, which has been freed from carbon dioxide, and add 2 drops of phenolphthalein solution. If the solution is colored pink, not more than 0.2 mil of tenth-normal acid should be required to discharge the color; should the solution be colorless after adding the phenolphthalein, not more than 0.2 mil of tenth-normal alkali should be required to produce a pink color.

CALCIUM HYDROXIDE

(Slaked Lime)



Mol. Wt. 74.09

Calcium hydroxide is a dry, white powder, slightly soluble in water, readily soluble in dilute hydrochloric or nitric acid. In the air it gradually takes up carbon dioxide, forming calcium carbonate. Its aqueous solution (lime water) also soon acquires a film of calcium carbonate upon its surface when exposed to the air.

Calcium hydroxide is used chiefly for the detection of alkalis in magnesium salts; in the extraction of alkaloids from drugs, particularly in the assay of opium; and as a neutralizing and alkalizing agent.

The reagent should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Carbonates	Trace
Matter Insoluble in Hydrochloric Acid.....	0.1000%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0858%
Aluminum (Al)	0.1500%

Methods of Testing

Carbonates, Matter Insoluble in Hydrochloric Acid.

—Mix 5 gm. with 30 mls of water and add 20 mls of hydrochloric acid. Not more than slight effervescence should occur. Filter off any insoluble matter and wash, ignite and weigh it. The weight should not exceed 0.005 gm.

Chlorides.—Mix 1 gm. with 15 mls of water, add 5 mls of nitric acid, filter if necessary, and add silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Mix 4 gm. with 30 mls of water, add 16 mls of hydrochloric acid and filter if necessary. To the solution add barium chloride solution, heat to boiling, allow it to stand twelve to eighteen hours and collect, wash, ignite and weigh any precipitate of barium sulphate. The weight should not exceed 0.01 gm.

Aluminum.—Dissolve 2.5 gm. in a slight excess of 5% hydrochloric acid and then add a slight excess of ammonia water. Not more than a slight flocculent precipitate should be produced.

CALCIUM OXIDE

(Lime)

CaO

Mol. Wt. 56.07

Calcium oxide is usually in the form of lumps or a granular powder, white or sometimes with a yellowish or

brownish tint due to constituents (iron) of the material used in its preparation. It is soluble in about 800 parts of water. Lime is generally made by ignition of marble or limestone. For special purposes it may be prepared by igniting egg shells or calcium salts, such as the nitrate, oxalate or carbonate.

Much heat is evolved on moistening the oxide with water, this giving evidence of its proper preparation and preservation. The addition of water forms calcium hydroxide, slaked lime, which when mixed with 3 or 4 parts of water, forms a smooth magma known as milk of lime. An approximately saturated solution is known as lime water.

Among other uses calcium oxide is employed in the extraction of alkaloids from drugs and in the determination of boric acid. Both alone and combined with sodium hydroxide as soda lime it is used as an absorbent for carbon dioxide and in estimating nitrogen, chlorine, sulphur, and phosphorus in organic compounds. In estimating small quantities of these, account must be taken of the presence of these elements in the reagent, as amounts are frequently present which, if disregarded, might vitiate results. Lime prepared from Iceland spar or from calcium salts is preferable in such cases, as it can usually be obtained practically free from the elements in question.

Lime must be stored in well-closed, air-tight containers owing to its affinity for water and carbon dioxide, and when desired particularly free from these substances it should be freshly ignited before use.

Maximum Limits of Impurities

Volatile Substances	10.0000%
Chlorides (Cl)	0.0050%
Silica (SiO_2)	0.1000%
Sulphates (SO_4)	0.0858%
Alumina (Al_2O_3)	0.2500%

Methods of Testing

Volatile Substances (Water, Carbon Dioxide, etc.).

—Ignite a portion of the calcium oxide to constant weight. The loss should not exceed 10 per cent.

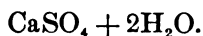
Chlorides.—Dissolve 1 gm. in 10 mls of nitric acid diluted with 10 mls of water and add silver nitrate solution. At most a slight opalescence should be produced.

Silica.—Slake 5 gm. with about 10 mls of water, add 25 mls of hydrochloric acid diluted with 25 mls of water and collect, wash, ignite and weigh any insoluble matter. Not over 0.005 gm. should be found.

Sulphates.—Slake 4 gm. with about 10 mls of water and add 20 mls of hydrochloric acid diluted with 25 mls of water. Filter if necessary, to the solution add barium chloride solution, heat to boiling and then allow to stand twelve to fifteen hours. Filter off, wash, ignite and weigh any precipitate of barium sulphate. Its weight should not exceed 0.01 gm.

Alumina.—Slake 2.5 gm. with 5 to 6 mls of water, dissolve the mass in a slight excess of 5% hydrochloric acid and then add a slight excess of ammonia water. Not more than a slight, flocculent precipitate should be produced.

CALCIUM SULPHATE



Mol. Wt. 172.16

Calcium sulphate generally occurs as a white powder or pulverulent pieces. It is soluble in about 400 parts of water, the solubility being increased by the presence of nitric or hydrochloric acid.

It is used in the main for the detection of barium and strontium, of oxalic acid in tartaric acid and of sodium carbonate in sodium bicarbonate; also for standardizing soap solution employed in water analysis.

Maximum Limits of Impurities

Insoluble Matter	0.0250%
Iron (Fe)	0.0040%
Magnesium and Alkalis	0.1000%
Chlorides (Cl)	0.0050%

Methods of Testing

Insoluble Matter (Barium Sulphate, Silica, etc.).—Dissolve 2 gm. in a mixture of 100 mls of water and 10 mls of hydrochloric acid with the aid of heat. Collect, wash and ignite any insoluble residue. Its weight should not exceed 0.0005 gm.

Iron.—To a solution of 2 gm., prepared as in the preceding test, add 25 mls of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should develop.

Magnesium and Alkalis.—Add an excess of ammonium oxalate solution to the mixture obtained in the test for iron, filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Chlorides.—Dissolve 1 gm. in a mixture of 15 mls of water and 15 mls of nitric acid with the aid of heat and add silver nitrate solution. Not more than a slight opalescence should develop.

CARBON DISULPHIDE

CS₂

Mol. Wt. 76.12

Carbon disulphide is a clear, colorless, or at most faintly yellowish liquid of specific gravity about 1.272 (theory 1.292 at 0°/4° C.), and boiling at 46° to 47° C. (theory 46.2° C.). It is practically insoluble in water (about 1 part in 500 at 0° C., less soluble at higher temperatures) but is miscible in all proportions with absolute alcohol or ether. Carbon disulphide is inflammable and cases of its spontaneous combustion when warm are known. Its vapor is also highly inflammable and with air forms an explosive mixture.

In pure condition carbon disulphide is colorless and has a rather pleasant ethereal odor, but it develops a disagreeable odor and a yellow color on exposure to light. The change is due to the formation of carbon monosulphide,

which sometimes separates as reddish-brown flocks, and free sulphur, which remains in solution but appears as a residue upon evaporation of the liquid.

Carbon disulphide is chiefly used in organic synthesis as a solvent for sulphur, iodine and other reagents; for the estimation of sulphur in ores and for removing sulphur from precipitated sulphides in analysis; in the electro-chemical deposition of silver; in testing cottonseed oil and tertiary alcohols; and in separating and detecting halogens.

The reagent should be stored in tightly-stoppered bottles in a cool place, remote from fire.

Maximum Limits of Impurities

Non-volatile Matter	0.0010%
Hydrogen Sulphide and Foreign Sulphur Compounds	0.0000%
Sulphuric and Sulphurous Acids	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 40 mls of carbon disulphide on the steam-bath. Not more than 0.0005 gm. of residue should remain.

Hydrogen Sulphide and Foreign Organic Sulphur Compounds.—Shake 10 mls of the carbon disulphide with about 0.5 gm. of lead carbonate. The latter should not acquire a brown color.

Shake 2 mls in a dry vessel with a globule of bright metallic mercury. The surface of the mercury should not become covered with a dark, pulverulent coating.

Sulphuric and Sulphurous Acids.—Shake 10 mls with 5 mls of water and test the latter with blue litmus paper. The paper should not be reddened or decolorized.

CARBON TETRACHLORIDE

CCl_4

Mol. Wt. 153.84

Carbon tetrachloride is a clear, colorless, non-inflammable liquid of specific gravity about 1.603 (theory 1.608 at

9.5°/4° C.) and boiling at 76° to 77° C. (theory 76.74° C.). It is soluble in about 1200 parts of water and is miscible in all proportions with absolute alcohol and with ether.

When pure, carbon tetrachloride has an agreeable ethereal odor, but technical grades often contain small amounts of sulphur compounds which impart a somewhat unpleasant odor. A method of determining carbon disulphide in carbon tetrachloride is given by Radcliffe, *J. Soc. Chem. Ind.*, **28**, 229 (1909); *abst.*, *J. Chem. Soc.*, **96**, II, 438 (1909).

Carbon tetrachloride is chiefly employed as a solvent for fats, resins, alkaloids, iodine, bromine, etc.; in the extraction and assay of drugs; in the analysis of mineral phosphates and ashes; in the toxicological detection of strychnine and atropine; and in the examination of hops, coffee and other materials.

The reagent should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Non-volatile Matter	0.0015%
Free Chlorine (Cl)	0.0002%
Hydrochloric Acid (HCl)	0.0010%
Organic Matter	0.0000%
Aldehyde	0.0000%
Carbon Disulphide (CS ₂)	0.0200%

Methods of Testing

Non-volatile Matter.—Evaporate 20 mls of carbon tetrachloride on the steam-bath. Not more than 0.0005 gm. of residue should remain.

Free Chlorine.—Shake 20 mls with 5 mls of zinc iodide-starch solution. No blue color should result.

Hydrochloric Acid.—Shake 20 mls with 10 mls of water for about one minute, remove the water and add to it silver nitrate solution. No change should be apparent.

Organic Matter.—Place 20 mls of carbon tetrachloride and 15 mls of sulphuric acid in a glass-stoppered cylinder which has previously been rinsed with sulphuric acid, and shake the mixture about every five minutes during one hour. The sulphuric acid should not become colored.

Aldehydes.—Mix 10 mls each of carbon tetrachloride and 30% potassium hydroxide solution and warm the mixture for one minute with frequent shaking. The potassium hydroxide solution should not acquire a yellow or brown color.

Carbon Disulphide.—Mix 10 mls each of carbon tetrachloride and a 10% solution of potassium hydroxide in absolute alcohol and allow the mixture to stand for one hour. Add 5 mls of 36% acetic acid and some copper sulphate solution and allow it to stand two hours longer. A yellow precipitate should not form.

CARMINE

Carmines is a red coloring matter derived from cochineal. It is usually in the form of bright red, light, friable pieces, soluble in ammonia water with a deep purplish-red color; insoluble in water or dilute acids.

Carmines is used principally in the preparation of carmine staining reagents, a list of which may be found in Gould's Illustrated Dictionary of Medicine, 5th ed., 1389-1400 (1907), together with a description of their methods of application.

The reagent should be stored in well-stoppered bottles and protected from light.

Maximum Limits of Impurities

Water	25.%
Ash	8.%
Matter Insoluble in Ammonia Water	Traces

Methods of Testing

Water.—Dry carmine at 100° C. The loss should not exceed 25%.

Ash.—Cautiously ignite approximately 0.25 gm., accurately weighed, in a porcelain crucible. The ash should not exceed 8% of the sample taken, and the odor of the

carmines while burning should resemble that obtained in the combustion of proteid. An odor of bromine or of phenol should not be apparent. These would indicate the presence of an eosine or pæonine lake, respectively, as an adulterant.

Matter Insoluble in Ammonia Water.—Dissolve 0.15 gm. in a mixture of 5 mls of ammonia water and 20 mls of water. Not more than a small amount of flocculent matter should remain undissolved.

CHLORAL HYDRATE

(Chloral Hydrate)



Mol. Wt. 165.4

Chloral hydrate is usually in the form of colorless crystals having an aromatic penetrating odor. It dissolves in about 0.5 part of water, 1.5 parts of alcohol or ether and in about 3 parts of chloroform. Exposed to the air it slowly volatilizes and when heated it melts at about 50° to 58° C. (theory 57° C.), the presence of traces of moisture considerably influencing the melting point.

Aqueous solutions of hydrated chloral, when freshly prepared, are neutral in reaction, but quickly acquire acidity due to decomposition with the formation of hydrochloric acid. Heat aids this decomposition and should be avoided in preparing its solutions. Alcoholic solutions also decompose gradually, chloral alcoholate being formed. With solutions of the caustic alkalis chloroform and a formate are produced.

Chloral hydrate is used chiefly as a reducing agent, especially in the determination of platinum (see the quantitative method under Platinum Chloride, page 239); for the detection of alpha and beta naphthols, phenetidin, myrrh, glycin, ammonia and hydrogen sulphide; and as a reagent in microscopy.

The reagent is affected by light and is easily discolored by organic matter (dust) and should therefore be stored in well-closed, amber-colored, glass-stoppered bottles.

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Hydrochloric Acid and Chlorides (Cl)	0.0020%
Organic Impurities	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the chloral hydrate and weigh the ash. Not more than 0.001 gm. should be found.

Hydrochloric Acid and Chlorides.—Dissolve 0.5 gm. in 10 mls of alcohol and add a few drops of silver nitrate solution. No change should be apparent at once.

Organic Impurities.—Place 0.5 gm. of the chloral hydrate and 5 mls of sulphuric acid in a clean glass-stoppered cylinder and shake the mixture frequently during half an hour. The acid should not become colored.

CHLORINE WATER

Chlorine water is a clear, pale greenish-yellow liquid having a strong odor of chlorine. It consists of a solution of chlorine in water and contains at least 0.4 gm. of Cl per 100 mls.

Chlorine water is made by passing chlorine gas into cold water, the maximum solubility being reached at about 10° C. The gas may be generated from manganese dioxide and hydrochloric acid and should be washed free from hydrochloric acid before absorption. Sometimes during absorption of the chlorine, especially at temperatures somewhat below 10° C., crystals of chlorine hydrate, HOCl, appear in the liquid; these crystals gradually disappear at ordinary temperature yielding up their chlorine again to the water.

Chlorine water is used mainly in the detection of iodine, bromine, quinine, xanthine and uric acid and as an oxidizing and bleaching agent. Under the best conditions of storage it is not a very stable preparation. Light and air aid in its decomposition with the formation of hydrochloric

acid and oxygen, while the chlorine gas readily escapes from the solution, necessitating its frequent renewal. It should be stored in small, well-filled and tightly-stoppered bottles in a cool, dark place.

Maximum Limits of Impurities

Non-volatile Matter	0.0025%
Hydrochloric Acid (HCl)	0.0180%

Methods of Testing

Non-volatile Matter.—Evaporate 20 mils of chlorine water on the steam-bath. Not more than 0.0005 gm. of residue should remain.

Hydrochloric Acid.—Shake vigorously 20 mils of chlorine water with about 2 mils of mercury for five minutes. Filter, add phenolphthalein to the filtrate and then titrate with normal alkali solution to the red end-point. Not more than 0.1 mil should be required.

Quantitative Method.—To a solution of about 1 gm. of potassium iodide in 25 mils of water add about 25 mils of the chlorine water, accurately measured. Titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 0.4 gm. of Cl per 100 mils of chlorine water should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.003546$ gm. of Cl,
log 54974.

CHLOROFORM

CHCl_3 ,

Mol. Wt. 119.39

Chloroform for reagent use is a clear, colorless, very volatile liquid, slightly soluble in water (about 300 parts) but miscible in all proportions with alcohol and ether. It has a specific gravity of 1.485 to 1.490 (theory 1.476 at 22° C.) and boils at 60° to 62° C. (theory 62° C.).

Chloroform ordinarily contains a little alcohol added as a preservative, the specific gravity limits here given insuring the presence of the proper amount for the purpose.

When a perfectly pure chloroform, free from alcohol, is required, it may be obtained by shaking the reagent with water, then separating the chloroform, mixing it with dry potassium carbonate and finally distilling. Chloroform free from alcohol is prone to decompose on exposure to light and air.

Besides being free from acidity as determined by the method of testing prescribed below, chloroform is sometimes required to be neutral to erythrosine for use in determining the alkalinity of water. It may be tested for suitability for this purpose by adding to 100 mls of water 5 mls of chloroform and 2.5 mls of a solution of 0.1 gm. of erythrosine (the sodium salt) in 1 liter of water and bringing the mixture to neutrality if necessary with fiftieth-normal sulphuric acid. The further addition of 5 or 10 mls of the chloroform should not disturb the end-point reached.

Besides its extensive employment for a variety of purposes dependent upon its well-known solvent properties, chloroform enters into some reactions as an essential reagent, such as the identification of primary amines and of naphthol.

Chloroform should be stored in a cool place in air-tight containers, best kept as nearly full as possible to avoid undue contact with air. Bottles should be of amber glass and glass-stoppered, sealed, if desired, with gelatin; sealing materials likely to contaminate the reagent, such as rosin, paraffin, etc., should not be used.

Maximum Limits of Impurities

Non-volatile Matter	0.0010%
Hydrochloric Acid (HCl)	0.0001%
Free Chlorine (Cl)	0.0002%
Phosgene	0.0000%
Aldehyde	0.0000%
Foreign Organic Bodies	0.0000%

Methods of Testing

Non-volatile Matter.—Evaporate 70 mls on the steam-bath. Not over 0.001 gm. of residue should remain.

Hydrochloric Acid.—Shake 20 mls of chloroform and 10 mls of water together for about one minute, allow the liquids to separate and draw off the water. It should not redden blue litmus paper. Add silver nitrate solution to the separated water. No change should be apparent.

Free Chlorine.—Shake 20 mls of chloroform with 5 mls of zinc iodide-starch solution. The latter should not become blue.

Phosgene.—Overlay 10 mls of chloroform with 'clear baryta water. No white film should form at the zone of contact of the liquids.

Aldehyde.—Warm 10 mls of chloroform with 10 mls of 30% solution of potassium hydroxide while stirring vigorously. No brown or yellow color should develop in the potassium hydroxide solution.

Foreign Organic Bodies.—Into a glass-stoppered flask, previously rinsed with sulphuric acid, put 20 mls of chloroform, 15 mls of sulphuric acid and 0.2 ml of formaldehyde (p. 160) and shake the mixture frequently during one-half hour. The acid should not acquire a color in this time.

CHROMIUM TRIOXIDE, FREE FROM SULPHATES

(Chromic Acid; Chromic Anhydride)

CrO_3

Mol. Wt. 100.0

Chromium trioxide usually occurs as dark brownish-red, deliquescent needles or prismatic crystals, soluble in less than 1 part of water. It contains at least 98% of CrO_3 .

Chromium trioxide in solid form or in solution, and especially when dissolved in sulphuric acid, is an energetic oxidizing agent; when mixed with strongly reducing substances it sometimes causes fire or explosion.

The reagent is used chiefly as an oxidizer; in the determination of carbon and phosphorus; in estimating sucrose; as a reagent for hydrogen peroxide, lactic and tartaric acids,

olive oil, acetanilid, acetphenetidin, thalline, salicin, guaiacol, albumin, silk, atropine, cocaine, nicotine, sparteine, ptomaines, erbium and didymium; and as a hardening agent in microscopy. Regarding the use of chromium trioxide in the determination of carbon in iron and steel see Treadwell-Hall's Analytical Chemistry, 4th ed., II, 399 (1915) or Scott's Standard Methods of Chemical Analysis, 2d ed. revised, 102 (1918).

The reagent should be stored in well-closed, glass-stoppered bottles, and should not be allowed to come in contact with organic matter.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0125%
Alkali Salts	1.0000%

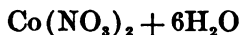
Methods of Testing

Sulphates.—Dissolve 10 gm. in 100 mils of water, add 20 mils of hydrochloric acid and some barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Alkali Salts (Chromates, Sulphates, etc.).—Ignite 0.2 gm. of the chromium trioxide in a porcelain dish, triturate the residue with 20 mils of water and filter. Evaporate the filtrate to dryness on a steam-bath, dry the residue at 100°C . and weigh it. The weight should not exceed 0.002 gm.

Quantitative Method.—Dissolve about 0.1 gm., accurately weighed, in 100 mils of water, add 5 gm. of potassium iodide, and 2 mils of hydrochloric acid, allow it to stand about ten minutes with frequent shaking, then dilute with about 200 mils of water and titrate the liberated iodine with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 98% of CrO_3 should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.003333 \text{ gm. of } \text{CrO}_3$,
log 52288.

COBALT NITRATE**(Cobaltous Nitrate)**

Mol. Wt. 291.09

Cobalt nitrate is usually in the form of red crystals, deliquescent in moist air and readily soluble in water or in alcohol (about 1 part of either). The salt melts if heated to 50° to 60° C.

The salt is used principally as a reagent for hydrogen peroxide; in differentiating citric, malic and tartaric acids; in blow-pipe analysis; for detecting aluminum, zinc and magnesium; for preparing sodium-cobaltic nitrite solution for detecting potassium; and as a catalyst.

The salt should be stored in tightly-stoppered bottles in a cool place.

Maximum Limits of Impurities

Sulphate (SO_4)	0.0100%
Chlorides (Cl)	0.0050%
Alkali Salts	0.2500%
Zinc (Zn)	0.0800%
Lead (Pb)	0.0200%
Copper (Cu)	0.0020%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 20 mls of water and add a few drops of hydrochloric acid and some barium chloride solution. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 2 to 3 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

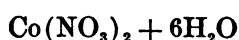
Alkali Salts.—Dissolve 2 gm. in 100 mls of water, make the solution ammoniacal and completely precipitate the cobalt by the addition of ammonium sulphide solution. Filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.005 gm.

Zinc.—Dissolve 0.5 gm. in 50 mls of water, add 5 mls of 27% sodium hydroxide solution, filter, to the filtrate add ammonium sulphide solution and allow it to stand ten minutes. The liquid should remain clear.

Lead and Copper.—Dissolve 2 gm. in 50 mls of water and add 2 mls of nitric acid and some hydrogen sulphide water. No change should be apparent.

COBALT NITRATE, FREE FROM NICKEL

(Cobaltous Nitrate)



Mol. Wt. 291.09

Cobalt nitrate, free from nickel, has the same appearance and properties as given under Cobalt Nitrate. The reagent may be used for the purposes enumerated under Cobalt Nitrate and for others requiring greater freedom from nickel. It should be stored in tightly-stoppered bottles in a cool place.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0100%
Chlorides (Cl)	0.0050%
Alkali Salts	0.2500%
Zinc (Zn)	0.1000%
Lead (Pb)	0.0200%
Copper (Cu)	0.0020%
Nickel (Ni)	0.0200%

Methods of Testing

Sulphates, Chlorides, Alkali Salts, Zinc, Lead and Copper.—Perform the tests as directed under Cobalt Nitrate. The results should be as there stated.

Nickel.—Dissolve 1 gm. in 20 mls of water, add 1.5 gm. of potassium or sodium cyanide and heat until a yellow color develops. Filter, cool, add 1 mil of bromine, shake until the bromine is dissolved and add 10 mls of 15% sodium hydroxide solution. No black precipitate should be produced.

COLLODION

Collodion is a colorless, or slightly yellowish, thick liquid, clear or at times very slightly opalescent. It is free from acidity and yields not less than 5% of residue upon evapora-

tion (pyroxylin). Collodion consists of a solution of pyroxylin in a mixture of ether and alcohol. When exposed in a thin layer a tough, transparent film is left after evaporation of the solvents.

Collodion is used as a reagent principally for differentiating phenol and creosote. It should be preserved in well-stoppered containers in a cool place, remote from fire.

Maximum Limit of Impurity

Acids 0.0000%

Methods of Testing

Acids.—Blue litmus paper moistened with water should not be reddened immediately when immersed in the collodion.

Determination of Residue.—To about 10 gm., accurately weighed and contained in a tared flask, add about 10 mls of water by drops with constant stirring. Evaporate the mixture and dry the residue at about 110° C. to constant weight. At least 5% of residue should remain.

CONGO RED

$(C_6H_4N:NC_{10}H_5NH_2SO_3Na)_2$

Mol. Wt. 696.52

Congo red is a brownish-red powder, readily soluble in warm water, yielding a blood-red solution; it is slightly soluble in cold water and in alcohol, insoluble in ether.

A solution suitable for use as an indicator is made by dissolving 0.5 gm. in a mixture of 90 mls of water and 10 mls of alcohol, 2 or 3 drops of this solution being usually sufficient for 100 mls of the solution to be titrated. Acids produce a violet color, alkalis restore the red color of the indicator.

Congo red is useful for estimating free mineral acids alone or in the presence of organic acids or inorganic salts which exhibit an acid reaction to other indicators. It is adapted to the titration of hydroxides and carbonates of

the alkalis and alkaline earths as well as of ammonia and amine bases, such as aniline and toluidine. For organic acids the indicator is useless. The presence of large quantities of neutral salts, such as the sulphates, chlorides and nitrates of the alkalis or alkali earths interferes with the end-point.

Aside from its use in ordinary acidimetric and alkali-metric titrations congo red is largely used in detecting and estimating hydrochloric acid in the gastric contents; in detecting acidity of papers; and as an addition to culture media in bacteriological investigations.

Method of Testing

Sensitiveness.—To 100 mls of water add 2 or 3 drops of the congo red solution described above. The red color of the liquid should be changed to reddish-violet on the addition of 1 drop of tenth-normal hydrochloric acid solution, and the addition of 1 drop of tenth-normal sodium hydroxide solution should restore the red color.

COPPER

Cu

At. Wt. 63.57

Copper occurs in various forms, such as foil, wire, turnings, filings and granules, adapted to various purposes. The metal generally preferred for reagent use is produced electrolytically, by which means a very pure product is obtained. Copper is a rather hard metal of characteristic yellowish-red color. It is readily soluble in nitric acid, less easily in hot, concentrated sulphuric acid and practically insoluble in hydrochloric acid. In dry air the metal undergoes no change but in moist air it gradually becomes covered with a green layer of basic copper carbonate. When heated, black cupric oxide forms upon its surface.

In the form of bright wire copper is used for the detection of mercury; in foil for the detection of arsenic by the Reinsch test; turnings are employed for the absorption of chlorine evolved in the combustion of organic substances in

iron and steel analysis; filings for the preparation of cuprous chloride solutions for gas analysis; and thin foil, wire or turnings for the determination of nitrogen in elementary analysis.

The reagent should be protected from contact with moist air, acid fumes, etc., by storage in well-stoppered bottles.

Maximum Limits of Impurities

Antimony and Tin	0.0000%
Lead	0.0000%
Silver	0.0000%
Iron and Bismuth (Fe_2O_3 , Bi_2O_3)	0.0500%
Arsenic (As)	0.0001%
Other Foreign Metals, Including Iron	0.1000%

Methods of Testing

Antimony and Tin.—Dissolve 10 gm. of copper in 60 mls of nitric acid, sp. gr. 1.3, evaporate the solution to dryness on the steam-bath and treat the residue with 50 mls of 10% nitric acid. Complete solution should result.

Lead.—To the solution obtained in the preceding test add 15 mls of sulphuric acid, evaporate on a sand-bath until fumes of sulphuric acid begin to be evolved and dissolve the residue in 100 mls of water. No insoluble residue of lead sulphate should remain.

Silver, Iron and Bismuth.—To the solution obtained in the test for lead add a few mls of hydrochloric acid. No turbidity should be produced. Then add ammonia water until the copper hydroxide first formed is redissolved, allow the mixture to stand three or four hours at about 50° C., filter and wash the filter paper free from copper with ammoniacal water. Dissolve any precipitate from the paper with a little dilute hydrochloric acid, wash with a little water, combine the acid solution and washings and again precipitate with ammonia water. Filter through the same paper as before, wash, ignite and weigh. The weight should not exceed 0.005 gm.

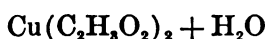
Arsenic.—Dissolve 10 gm. of copper in 60 mls of nitric acid, add to the solution 15 mls of sulphuric acid, evaporate

on a sand-bath until fumes of sulphuric acid begin to come off, dissolve the residue in 100 mls of water, introduce the solution into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

Other Foreign Metals, Including Iron.—Dissolve 10 gm. of copper in 60 mls of nitric acid, sp. gr. 1.3, add 15 mls of sulphuric acid, evaporate the solution on a sand-bath until sulphuric acid vapors begin to come off and dissolve the residue in 300 mls of water. Heat the solution to about 70° C., completely precipitate the copper by passing in hydrogen sulphide gas, filter, evaporate the filtrate, finishing on a sand-bath to expel sulphuric acid, ignite and weigh the residue. The weight should not exceed 0.01 gm.

COPPER ACETATE

(Cupric Acetate)



Mol. Wt. 199.65

Copper acetate usually occurs as dark bluish-green crystals, slightly efflorescent in dry air. It is soluble in about 8 parts of water and is also somewhat soluble in alcohol. Upon heating the salt or evaporating its aqueous solution, an insoluble or difficultly soluble basic salt is formed.

The reagent is used chiefly in the detection of glucose, colophony, cystin, resins, terpenes, carbon disulphide and artificial coloring of wines. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Insoluble Matter	Trace
Sulphates (SO_4)	0.0100%
Chlorides (Cl)	0.0100%
Salts of the Alkalis, Earths, etc.	0.2000%
Iron (Fe)	0.0175%

Methods of Testing

Insoluble Matter.—Powder 1 gm. of the copper acetate and dissolve it by agitation in 20 mls of cold water. The solution should not be more than slightly turbid.

Sulphates.—Dissolve 1 gm. in 20 mls of water acidulated with 1 mil of hydrochloric acid and add barium chloride solution. No turbidity should result.

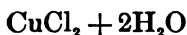
Chlorides.—Dissolve 0.5 gm. in 20 mls of water acidulated with 2 mls of nitric acid and add silver nitrate solution. No more than a slight opalescence should be produced.

Salts of the Alkalis, Earths, etc.—Perform these tests as directed under Copper Sulphate. The results should be as there stated.

Iron.—Perform the test as directed under Copper Sulphate, using 2 gm. instead of 5 gm. as a sample. The weight of the residue obtained should not exceed 0.0005 gm.

COPPER CHLORIDE, CUPRIC

(Copper Dichloride)



Mol. Wt. 170.52

Cupric chloride occurs usually as green crystals, deliquescent in moist air and efflorescent in dry air. It is soluble in about 2 parts of water or alcohol.

Cupric chloride is used chiefly in the determination of carbon in iron; as a substitute for ferric chloride in the analysis of iron containing sulphur and phosphorus; for the detection of gold-plated metal; and for the purification of alloys, especially from arsenic and antimony.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Substances Insoluble in Alcohol	0.0000%
Sulphates (SO ₄)	0.0100%
Salts of the Alkalis	0.0067%
Arsenic (As)	0.0010%
Iron (Fe)	0.0280%

Methods of Testing

Substances Insoluble in Alcohol.—Dissolve 5 gm. in 10 mls of water and mix the solution with 10 mls of alcohol. No turbidity should result.

Sulphates.—Dissolve 1 gm. in 20 mls of water, add 1 mil of hydrochloric acid and some barium chloride solution and allow to stand 5 minutes. No turbidity should result.

Salts of the Alkalis.—Dissolve 3 gm. in 100 mls of water, add 1 or 2 mls of hydrochloric acid, warm the solution to about 70° C., pass in hydrogen sulphide gas until the copper is completely precipitated, filter, evaporate the filtrate to dryness and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Arsenic.—Dissolve 1 gm. in 20 mls of water, introduce the solution a little at a time into a Marsh apparatus and allow the action to proceed for one hour. No deposit should form in the reduction tube.

Iron.—Dissolve 5 gm. in 25 mls of water, add 2 mls of nitric acid, heat to boiling and add 35 mls of ammonia water. Filter and wash the filter with ammoniacal water until the copper is all removed from the paper. Dissolve any precipitate remaining on the filter in a little hydrochloric acid and wash it from the filter. Collect and mix this acid solution and the washings and treat the mixture with ammonia water. Collect, wash, ignite and weigh any precipitate which may form. The weight should not exceed 0.002 gm.

COPPER CHLORIDE, CUPROUS

(Copper Monochloride)

CuCl

Mol. Wt. 99.03

Cuprous chloride is a white, or very nearly white, crystalline powder, insoluble in water but soluble in hydrochloric acid or in ammonia water.

Exposed to the air the salt becomes green, due to oxidation; the change takes place more readily when moisture is present. Light also affects the color of cuprous chloride, producing bluish tints; direct sunlight blackens it. The purity of the preparation may be judged from its appear-

ance. It should be white or very nearly so, but not green or brown.

The reagent is used chiefly in gas analysis for absorption of carbon monoxide, arsine, stibine and some gaseous hydrocarbons.

Cuprous chloride should be stored in tightly-stoppered bottles, protected from light. When perfectly dry, changes due to air and light are very slow. Metallic copper, in wire or strips, should be added to its solutions, both acid and ammoniacal, to prevent their oxidation.

COPPER OXIDE

(Cupric Oxide)

CuO

Mol. Wt. 79.57

Copper oxide occurs as coarse granules, wire or powder. It is readily soluble in dilute acids, slowly in ammonia water or ammonium carbonate solution. Copper oxide, especially when heated, is reduced to metallic copper by many substances, such as hydrogen, carbon monoxide, methane and hydrazin; sometimes with explosive violence, as by metallic magnesium. Upon this property depends its most general uses.

The reagent is used principally in ultimate analysis for the determination of hydrogen and carbon; in blowpipe analysis for the detection of chlorine, bromine and iodine; in gas analysis in determining hydrogen and hydrocarbons; as a catalyst; and as a cathode in certain electrolytic preparations.

I

COPPER OXIDE, GRANULAR

Copper oxide, granular, is usually in the form of coarse, grayish-black granules.

Maximum Limits of Impurities

Nitrates and Carbonates	0.0000%
Calcium (Ca)	0.0013%
Sulphates (SO ₄)	0.1000%
Chlorides (Cl)	0.0050%

Methods of Testing

Nitrates, Carbonates, Calcium and Chlorides.—Perform these tests as directed under Copper Oxide, Powder. The results should be as there stated. Powder the copper oxide before applying the tests for calcium and chlorides.

Sulphates.—Dissolve 1 gm. in 5 mls of hydrochloric acid, dilute with 20 mls of water and add barium chloride solution. Not more than a slight turbidity should be produced.

II

COPPER OXIDE, POWDER

Copper oxide, powder, occurs as a fine, black, amorphous powder.

Maximum Limits of Impurities

Nitrates and Carbonates	0.0000%
Chlorides (Cl)	0.0050%
Acid-insoluble Matter	0.0500%
Substances not Precipitated by Hydrogen Sulphide.	0.2500%
Water-soluble Substances	0.0167%
Sulphates (SO ₃)	0.0400%
Calcium (Ca)	0.0013%

Methods of Testing

Nitrates and Carbonates.—Heat 100 gm. of copper oxide in a glass tube, pass over it a stream of moist air, freed from carbon dioxide, and test the vapors with moist blue litmus paper and also by passing them into lime water. The paper should not be reddened and the lime water should not be rendered turbid.

Chlorides.—Dissolve 1 gm. in a mixture of 10 mls of nitric acid and 10 mls of water, and add silver nitrate solution. The solution should not become cloudy.

Acid-insoluble Matter and Substances not Precipitated by Hydrogen Sulphide (Iron, etc.).—Dissolve 2 gm. in 10 mls of hydrochloric acid, dilute with 100 mls of water and collect and weigh any insoluble matter. The weight should not exceed 0.001 gm. Heat the solution to

about 70° C., pass in hydrogen sulphide until the copper is completely precipitated, filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.005 gm.

Water-soluble Substances.—Shake 3 gm. with 60 mls of water for a few minutes, filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.0005 gm.

Sulphates.—Dissolve 1 gm. in 4 mls of hydrochloric acid, dilute with 20 mls of water and add barium chloride solution. No change should be apparent.

Calcium.—Digest 20 gm. with a mixture of 5 mls of nitric acid and 100 mls of water for about fifteen minutes, shaking frequently. Filter, precipitate the copper from the filtrate completely by means of hydrogen sulphide gas and filter again. Evaporate the filtrate on the steam-bath to about 20 mls, add an excess of ammonia water, filter once more and to the filtrate add ammonium oxalate solution. No precipitate should be produced at once.

III

COPPER OXIDE, WIRE

Copper oxide, wire, usually occurs as short, cylindrical, black pieces, having the form of the wire from which they were made.

Maximum Limits of Impurities

Nitrates and Carbonates	0.0000%
Chlorides (Cl)	0.0050%
Calcium (Ca)	0.0013%
Sulphates (SO ₄)	0.0400%
Acid-insoluble Matter	0.0500%
Substances not Precipitated by Hydrogen Sulphide.	0.2500%

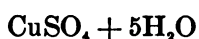
Methods of Testing

Nitrates, Carbonates, Chlorides, Calcium, Sulphates, Acid-insoluble Matter (Metallic Copper, etc.)

and Substances not Precipitated by Hydrogen Sulphide.—Perform the tests as directed under Copper Oxide, Powder. The results should be as there stated.

COPPER SULPHATE

(Cupric Sulphate)



Mol. Wt. 249.71

Copper sulphate occurs usually as blue, transparent crystals, sometimes as small granules, slightly efflorescent in dry air. It is soluble in about 3 parts of water but is almost insoluble in alcohol. The aqueous solution is acid to litmus.

Copper sulphate is used principally for preparing various reagents used in the detection and determination of reducing sugars; as a reagent for peptone, albumin, urea, aloes and picric acid; in testing for iodine and carbon disulphide; in determining urobilin in urine and estimating sodium hydrosulphite.

The salt should be stored in well-stoppered bottles:

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Salts of the Alkalis, Earths, etc.	0.2500%
Iron (Fe)	0.0140%

Methods of Testing

Insoluble Matter.—Dissolve 5 gm. in 50 mls of water. The solution should be clear and free from undissolved residue.

Salts of the Alkalis, Earths, etc.—Dissolve 2 gm. in 100 mls of water, add 2 mls of hydrochloric acid, heat the solution to 70° C., pass in hydrogen sulphide gas until the copper is completely precipitated, filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.005 gm.

Iron.—Dissolve 5 gm. in 25 mls of water, add 2 mls of nitric acid, heat to boiling, add 20 mls of ammonia water,

and filter the mixture through an ashless filter. Wash the filter with ammoniacal water until perfectly free from copper, dissolve any residue on the filter in a little dilute hydrochloric acid, wash the paper with water, mix the washings with the acid solution and again treat with an excess of ammonia water. Filter through the same paper, wash the paper with a little water, incinerate it and ignite and weigh any precipitate it may contain. The weight should not exceed 0.001 gm.

COPPER AND AMMONIUM CHLORIDE

(Ammonio-Cupric Chloride)



Mol. Wt. 277.51

Copper and ammonium chloride usually occurs as blue crystals, soluble in about 4 parts of water, yielding a solution slightly acid to litmus. The salt is also soluble in alcohol.

Solutions of the reagent readily dissolve metallic iron and find their principal use in the determination of carbon in iron and steel.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Free Acids	0.0000%
Sulphates (SO_4)	0.0100%
Salts of the Alkalis, Earths, etc.	0.2000%
Iron (Fe)	0.0140%

Methods of Testing

Insoluble Matter, Free Acids.—Dissolve 30 gm. in 100 mls of water. The solution should be clear. Introduce into the solution several pieces of piano wire. The wire should dissolve with the deposition of copper but without any apparent evolution of gas.

Sulphates.—Dissolve 1 gm. in 20 mls of water, add 1 mil of hydrochloric acid and some barium chloride solution and allow it to stand five minutes. No change should be apparent.

Salts of the Alkalies, Earths, etc., and Iron.—Perform the tests as directed under Copper Sulphate. The results should be as there stated.

DEVARDA'S METAL

(Devarda's Alloy)

Devarda's metal occurs usually as a gray powder and is composed of 50 parts of copper, 45 parts of aluminum and 5 parts of zinc. It is partly soluble in hydrochloric acid, the copper remaining undissolved. The reagent is employed in the determination of nitrogen by the reduction of nitrates, nitrites, etc., to ammonia.

Maximum Limit of Impurity

Nitrogen (N) 0.0056%

Method of Testing

Nitrogen.—Place 10 gm. of Devarda's metal in a flask of about 1 liter capacity, add 100 mls of hydrochloric acid and allow to stand with frequent agitation until hydrogen ceases to be evolved. Cool the mixture, connect the flask with a condenser, add 100 mls of 27% solution of sodium hydroxide and distil 50 to 75 mls, collecting the distillate in 10 mls of tenth-normal hydrochloric acid solution. Titrate the excess acid in the distillate with tenth-normal solution of sodium hydroxide. Not more than 0.4 ml of the tenth-normal acid solution should be neutralized by the ammonia in the distillate.

DIMETHYLAMINOAZOBENZENE

(Butter Yellow)



Mol. Wt. 225.21

Dimethylaminoazobenzene is usually in the form of yellow, crystalline plates, melting between 115° and 117° C. (theory 115° C.). It is insoluble in water but is readily soluble in alcohol.

As an indicator dimethylaminoazobenzene somewhat resembles methyl orange. In acid and alkaline solutions its color is red and yellow respectively, and it is not affected by carbonic acid. It gives a sharp color change with mineral acids but is useless for titrating organic acids or acid salts. An indicator is made by dissolving 0.5 gm. in 100 mls of alcohol. This solution is commonly known as Toepfer's reagent.

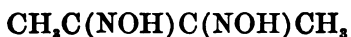
Dimethylaminoazobenzene is principally used in the determination of free hydrochloric acid in gastric juice; and also for the microscopical identification of fat.

Method of Testing

Sensitiveness.—Add 1 or 2 drops of the alcoholic solution above described to 25 mls of water. The bright lemon-yellow color imparted to the liquid should be changed to orange-red by 1 drop of tenth-normal hydrochloric acid solution and on adding 1 drop of tenth-normal sodium hydroxide solution the yellow color should be restored.

DIMETHYLGLYOXIME

(Diacetyldioxime)



Mol. Wt. 116.10

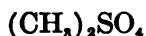
Dimethylglyoxime is usually obtained as white, needle-shaped crystals or a crystalline powder, soluble in alcohol but almost insoluble in cold water and very difficultly soluble in boiling water. It melts at about 240° C. (theory 232° to 233° C.).

Dimethylglyoxime is a very important reagent for the determination of nickel and its adaptability to other purposes has of late been under investigation. It has been recommended for the separation of nickel from cobalt and from palladium and for separating palladium from tin, gold, platinum, rhodium and iridium. It is also used for detecting ferrous salts. In determining nickel the precipitate of nickel dimethylglyoxime may be weighed or it may be redissolved and the nickel determined by electrolysis.

Method of Testing

Suitability as a Reagent for Nickel.—Dissolve 2.4 gm. of cobalt-free nickel chloride ($\text{NiCl}_2 + 6\text{H}_2\text{O}$), or 2.9 gm. of cobalt-free nickel nitrate [$\text{Ni}(\text{NO}_3)_2 + 6\text{H}_2\text{O}$], in water and dilute to 100 mls. Dilute 10 mls of this solution to 100 mls, heat to boiling and add a solution of 0.25 gm. of dimethylglyoxime in 25 mls of alcohol. Add ammonia water by drops until the mixture is alkaline; cool and filter. Heat the filtrate to boiling with more of the alcoholic dimethylglyoxime solution. No brown color should develop and no red precipitate should be formed on cooling again.

DIMETHYL SULPHATE



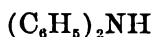
Mol. Wt. 126.12

Dimethyl sulphate is a clear, colorless, oily liquid. It is readily soluble in alcohol and in ether, but is almost insoluble in water. It has a specific gravity of about 1.333 (theory 1.33344 at 15° C.) and boils at about 188° C. (theory 188.3°–188.6° C.).

Dimethyl sulphate is used principally for testing creosote oils and creosote dips for mineral oils and creosote of doubtful origin; for detecting tar oils in mineral oils and printers ink; and in organic synthesis. Regarding the testing of creosote oils and dips see Circular No. 167 (1911), Bureau of Animal Industry, U. S. Dept. of Agriculture.

The reagent should be stored in well-closed, glass-stoppered bottles. It should be handled with great care as it is extremely caustic and its vapors are poisonous.

DIPHENYLAMINE



Mol. Wt. 169.16

Diphenylamine usually occurs as white crystals, having a slight aromatic odor. The reagent is only slightly soluble in water, but readily soluble in alcohol or ether. It melts at about 53° C. (theory 52.85° C.) and boils at a little above 300° C. (theory 310° C.).

Diphenylamine is weakly basic in character and dissolves in acids more readily than in water. A solution of 0.5 gm. in a mixture of 100 mls of sulphuric acid and 20 mls of water is much used as a qualitative test-solution for nitrates. When used so as to form a layer above or below the liquid tested, according to the specific gravity of the latter, it constitutes a test of extreme sensitiveness. Other oxidizing agents, such as chlorine, peroxides, chromates and ferric salts, should be absent when testing for nitric acid with diphenylamine.

The reagent is used principally for detecting nitrates, wood fiber in paper, and sugar in urine.

Maximum Limits of Impurities

Nitrates (N_2O_5)	0.0150%
Aniline	0.0500%

Methods of Testing

Nitrates.—Dissolve 0.2 gm. in 20 mls of sulphuric acid mixed with 2 mls of water. The solution should be colorless. The sulphuric acid used should be free from nitric acid.

Aniline.—Powder 1 gm. of the diphenylamine and pour the powder into 20 mls of a solution of chlorinated lime. A violet color should not develop.

ETHER, Sp. Gr. 0.725

(Ethyl Ether)

$(\text{C}_2\text{H}_5)_2\text{O}$

Mol. Wt. 74.1

Ether, sp. gr. 0.725, is a clear, colorless, very volatile and inflammable liquid, consisting of about 96% of $(\text{C}_2\text{H}_5)_2\text{O}$, and about 4% of alcohol containing a little water. It has a specific gravity not above 0.726 and boils at about 35°C . (theory 34.97°C). It is miscible with alcohol, chloroform benzene and petroleum benzin. The vapor of ether is

highly inflammable and with air forms an explosive mixture.

Under the action of air, light and moisture ether is gradually oxidized with the production of a number of bodies, such as aldehydes, acetic acid, vinyl alcohol, hydrogen peroxide, etc. Ethyl peroxide formed in this way may be left as a residue upon evaporation and may explode violently with heat or percussion.

Ether, sp. gr. 0.725, is employed as a solvent for fats, resins, alkaloids and a variety of other substances. The reagent should be stored in well-stoppered bottles of amber glass in a cool, dark place, remote from fire. Owing to the high coefficient of expansion of ether vapor the bottles should contain ample air space.

Maximum Limits of Impurities

Residue on Evaporation	0.0024%
Acids	0.0000%
Aldehydes	0.0000%
Peroxides	0.0000%

Methods of Testing

Residue on Evaporation.—Evaporate 50 mls on a steam-bath, dry the residue at 100° C. and weigh it. Not more than 0.001 gm. should be found.

Acids.—Immerse in the ether blue litmus paper, previously moistened with water, and allow to remain ten minutes. The paper should not be reddened.

Aldehydes.—Shake 10 mls of ether occasionally during one hour with 1 mil of 5% solution of potassium hydroxide in a glass-stoppered cylinder, protected from light. No color should develop in either liquid.

Peroxides.—Shake 10 mls of ether occasionally during one hour with 1 mil of freshly prepared 10% solution of cadmium and potassium iodide in a glass-stoppered cylinder, protected from light. No color should develop in either liquid.

ETHER, ANHYDROUS, DISTILLED OVER SODIUM

 $(C_2H_5)_2O$

Mol. Wt. 74.1

Ether, anhydrous, distilled over sodium, is a clear, colorless liquid having a specific gravity of 0.720 (theory 0.7111 at 22° C.). In other respects it has the same general properties described under Ether, Sp. Gr. 0.725.

This reagent is employed for the same purposes as Ether, Sp. Gr. 0.725, and particularly in those cases requiring absence of water and alcohol. The conditions of storage prescribed under Ether, Sp. Gr. 0.725, apply also to this article.

Maximum Limits of Impurities

Residue on Evaporation	0.0024%
Peroxides	0.0000%
Acids	0.0000%
Aldehydes and Vinyl Alcohol	0.0000%
Sulphur Compounds	0.0000%
Acetone	0.0050%
Water	0.1000%
Alcohol	0.3000%

Methods of Testing

Residue on Evaporation, Peroxides and Acids.—Perform these tests as given under Ether, Sp. Gr. 0.725. The results should be as there stated.

Aldehydes and Vinyl Alcohol.—Shake 20 mls of the ether with 5 mls of Nessler's solution for one minute in a glass-stoppered flask. No reddish-brown or black precipitate should be produced.

Sulphur Compounds.—Shake 20 mls of the ether with a globule of mercury for two minutes in a glass-stoppered flask. No black precipitate should form and the mercury should retain its luster.

Acetone.—Shake 6 mls of barium hydroxide solution and 2 mls of 5% solution of mercuric chloride with 20 mls of the ether for one minute. Allow the liquids to separate, filter the aqueous portion and to the clear filtrate add am-

monium sulphide solution. No dark color should appear within ten minutes.

Water and Alcohol.—Place 15 mls of the ether in a dry, glass-stoppered flask with a freshly cut piece of sodium about the size of a pea, and allow it to stand three to four hours. Only a slight evolution of gas should take place and the sodium should not become covered with a white coating, but should retain its metallic appearance.

FORMALDEHYDE, NEUTRAL, 37%

(Formic Aldehyde)

HCHO

Mol. Wt. 30.02

Formaldehyde, neutral, 37%, is a clear, colorless liquid of pungent odor. It is clearly miscible with water or alcohol and is neutral to litmus paper. It contains at least 37% of HCHO.

Formaldehyde on long standing, especially in the cold, becomes cloudy due to the separation of paraformaldehyde. The reagent contains a varying amount of methyl alcohol, the presence of which tends to retard this polymerization. Exposed to air and light it acquires acidity due to formic acid.

Formaldehyde, neutral, is particularly useful for making colloidal gold solutions;* it is also employed in the detection of boiled milk, peppermint and sesame oils, phenolic derivatives, glucose, hydrogen peroxide, blood, various alkaloids and many other substances; as a reducing agent in the de-

* Regarding the preparation and use of colloidal gold solutions see Kaplan and McClelland, *J. Amer. Med. Assoc.*, 62, 511 (1914); *abst.*, *Chem. Abst.*, 8, 1596 (1914). Rubenstone and Schwartz, *N. Y. Med. Jour.*, 101, 1273 (1915). Miller, Brush, Hammers and Felton, *Bull. Johns Hopkins Hosp.*, 26, 298 (1915); *abst.*, *Chem. Abst.*, 10, 912 (1916). Cummer, *Cleveland Med. Jour.*, 16, 813 (1917). Lee, *St. Luke's Hospital Med. and Surg. Report*, 4, 216 (1917); *abst.*, *Chem. Abst.*, 11, 2913 (1917). Black, Rosenberg and McBride, *J. Amer. Med. Assoc.*, 69, 1855 (1917); *abst.*, *Chem. Abst.*, 12, 377 (1918). Speidel and Smith, *U. S. Naval Med. Bull.*, 12, 220 (1918); *abst.*, *Chem. Abst.*, 12, 1686 (1918).

termination of gold, mercury and other metals; and in the titration of ammonium citrate and of amino-nitrogen in blood.

Formaldehyde should be stored in well-stoppered bottles of amber glass in a moderately warm place.

Method of Testing

Quantitative Method.—Tare a flask containing about 10 mls of water, introduce about 3 mls of the formaldehyde and weigh accurately. Add 50 mls of normal sodium hydroxide solution followed immediately but slowly through a small funnel with 50 mls of 3% solution of hydrogen peroxide, previously rendered neutral to litmus. Heat the mixture on a steam-bath for five minutes with occasional shaking, allow it to cool, rinse the funnel and sides of the flask, let it stand thirty minutes and titrate with normal sulphuric acid solution, using litmus as indicator. Not less than 37% of HCHO should be found.

1 mil of normal NaOH = 0.03002 gm. of HCHO, log 47741.

FURFURAL

(Furfurol)

$C_5H_4O_2$

Mol. Wt. 96.06

Furfural is a clear, pleasant smelling liquid, usually having a yellow color which it acquires on exposure to air and light. When freshly prepared it is colorless. Furfural has a specific gravity of 1.165 to 1.166 (theory 1.1594 at 20°/4° C.) and boils at 158° to 160° C. (theory 161° C.). It is soluble in about 15 parts of water and is easily miscible with alcohol and ether.

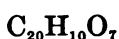
Furfural is used principally in the detection of sesame oil in margarin butter and margarin cheese and as a reagent for urea, alkaloids, iso-amyl alcohol, santonin, cholesterin, biliary acids, acetone, phenol, skatol and tyrosin.

The reagent should be stored in well-stoppered bottles or sealed tubes, protected from light. It is often obtained

in sealed tubes in which the air has been replaced by hydrogen and in which it keeps well. In many cases a yellow or light brown color does not render it unsuitable for use.

GALLEIN

(Pyrogallolphthalein)



Mol. Wt. 362.18

Gallein usually occurs as a brown powder, sometimes as greenish, lustrous crystals. It is almost insoluble in water but is soluble in alcohol. An indicator solution may be prepared by dissolving 0.5 gm. in 100 mls of 50% alcohol; 2 or 3 drops of this solution are added to about 100 mls of the solution to be titrated.

Gallein is useful in titrating mineral acids, organic acids, alkali hydroxides, ammonia and alkaloids but is useless for carbonates. It is more sensitive to alkalis than is phenolphthalein, and may be used in the presence of ammonium salts. It is recommended for determining phosphates in urine, monobasic phosphates giving with it a yellow color; dibasic, red; and tribasic, violet. The sensitiveness of the indicator is said to be increased by the presence of alcohol or ether. Color changes alter somewhat on standing so that readings should be made at once.

GLYCERIN

(Glycerol)



Mol. Wt. 92.08

Glycerin is a clear, colorless, or very nearly colorless, syrupy, almost odorless liquid having a sweet taste. It is miscible with water and alcohol, but not with chloroform, ether, benzene or carbon disulphide. It has a specific gravity not below 1.25 and its 10% aqueous solution is neutral to litmus paper. It is hygroscopic.

Glycerin is used chiefly in testing creosote, catechu and extract of malefern; in detecting albumin in urine; and in

the titration of boric acid. It is a constituent of several reagents for detecting and determining glucose.

The reagent should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Inorganic Matter	0.0080%
Chlorides (Cl)	0.0008%
Suphates (SO ₃)	0.0015%
Oxalic Acid (H ₂ C ₂ O ₄)	0.0065%
Calcium (Ca)	0.0050%
Heavy Metals	0.0000%
Arsenic (As)	0.0012%
Readily Carbonizable Matter	0.0000%
Sugars (as Saccharose)	0.0400%
Ammonium Compounds (NH ₃)	0.0030%
Fatty Acid Esters (as Ethyl Butyrate)	0.1161%
Substances Reducing Ammoniacal Silver Nitrate Solution	0.0000%
Dextrose and Acrolein	0.0000%

Methods of Testing

Inorganic Matter.—Heat 10 mils of glycerin in an open dish, ignite the vapors, and when the glycerin has been entirely consumed, ignite any residue at a low red heat and weigh the ash. The weight should not exceed 0.001 gm.

Chlorides.—Dilute 5 mils with 25 mils of water and add silver nitrate solution. Not more than a slight opalescence should result.

Sulphates.—Dilute 5 mils with 25 mils of water and add barium chloride solution. No change should be apparent.

Oxalic Acid.—Dilute 5 mils with 25 mils of water and add calcium chloride solution. No change should be apparent.

Calcium.—Dilute 5 mils with 25 mils of water and add ammonium oxalate solution. No change should be apparent.

Heavy Metals.—Dilute 5 mils with 25 mils of water and add hydrogen sulphide water. No change should be apparent.

Arsenic.—Mix 1 mil with 3 mils of stannous chloride solution and let stand one hour. No darkening of the mixture should take place.

Readily Carbonizable Matter.—Mix 5 mils with 5 mils of sulphuric acid and allow to stand one hour. The mixture should not become darker than yellow.

Sugars.—Mix 5 mils of glycerin, 5 mils of water and 5 drops of hydrochloric acid, warm the mixture a few minutes on a steam-bath, add 3 drops of copper sulphate solution and 1 mil of 27% solution of sodium hydroxide and warm a few minutes more on the steam-bath. No separation of cuprous oxide should be visible.

Ammonium Compounds.—Mix 1 mil with 1 mil of sodium hydroxide solution and warm the mixture on the steam-bath. The vapors should not turn moistened red litmus paper blue.

Fatty Acid Esters.—Mix 40 mils with 50 mils of freshly boiled water and 2.5 mils of normal sodium hydroxide solution and boil the mixture for five minutes. Cool it and neutralize with normal hydrochloric acid solution, using phenolphthalein as indicator. Not less than 2 mils of the hydrochloric acid solution should be required.

Substances Reducing Ammoniacal Silver Nitrate Solution.—Mix 1 mil of glycerin and 1 mil of ammonia water, heat the mixture to about 60° C., add 3 drops of silver nitrate solution and allow to stand for five minutes. No coloration or brownish-black precipitate should develop.

Dextrose and Acrolein.—Heat 5 mils of glycerin with 5 mils of 10% solution of sodium hydroxide at 60° C. for 5 minutes. The mixture should not become yellow.

GOLD CHLORIDE

(Gold Chloride, Acid; Chlorauric Acid)



Mol. Wt. 394.1

Gold chloride is usually in the form of bright, golden-yellow crystals, very easily soluble in water; also soluble in

alcohol and in ether. It is very hygroscopic and deliquescent. The salt contains 48.5% to 50% of Au (theory 50.04%). Its solutions in alcohol or in ether on standing undergo decomposition with gradual reduction and separation of the gold.

Gold chloride is used mainly for the detection of glucose, indican, sulphocyanates, mercury and of cottonseed oil in olive oil; as an alkaloidal reagent; in testing suprarenal gland; and in preparing colloidal gold solution. In reference to the last named use see the footnote on page 160 under Formaldehyde, Neutral, 37%.

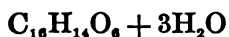
Gold chloride should be kept in sealed glass tubes or in well-closed, glass-stoppered bottles.

Method of Testing

Quantitative Method.—Dissolve 0.2 to 0.3 gm. of the salt, accurately weighed, in water, add 15 mls of solution of sodium sulphide (sp. gr. about 1.18) and dilute to 125 mls. Warm this solution to about 70° C. and electrolyze with a current of about 0.2 ampere and 2 to 3 volts, using a hollow conical or cylindrical cathode and spiral anode. After the gold is all removed from the solution (which generally takes two or three hours) remove the cathode, wash with water, dry at about 100° C. and weigh. The gain in weight of the cathode should correspond to 48.5% to 50% of Au.

The gold may also be precipitated in the metallic state from aqueous solutions of the gold chloride by warming with the addition of oxalic acid or by adding hydrogen peroxide to the alkalized solution.

HEMATOXYLIN



Mol. Wt. 356.24

Hematoxylin is usually in the form of colorless or yellowish crystals, slightly soluble in water or in ether, more readily in solutions of borax and easily in alcohol. In ammonia water it dissolves yielding a purple solution. On exposure to air and light it darkens, due to oxidation to

hematein. It is also very sensitive to ammonia vapor, which discolors it.

The reagent is used principally as an indicator, particularly in titrating alkaloids; as a reagent for iron and copper; and as a stain in microscopy. The test solution employed as an indicator contains 0.02 gm. in 10 mls of alcohol. About 5 drops of the solution are used in each titration. The indicator is yellow in acid solutions and violet to purple in alkaline. It is not well adapted to ordinary alkalimetric determinations, however.

Hematoxylin or its solutions should be stored in well-stoppered bottles protected from light.

HIDE POWDER

Hide powder is a white powder of woolly texture, having a slight but not disagreeable odor. It is used for the determination of tannins and non-tannins and, according to the official standards of the American Leather Chemists' Association,* should be well delimed and have acidity equivalent to 12 to 13 mls of tenth-normal sodium hydroxide solution for each 10 gm. of the dry powder.

Method of Testing

Acidity.—To 10 gm. of the hide powder add 100 mls of boiling water and stir the mixture for two or three minutes until the powder becomes well soaked and partly gelatinized. Then add 3 to 4 mls of phenolphthalein solution and titrate the mixture while still hot with tenth-normal sodium hydroxide solution, stirring well meanwhile, until a permanent pink color is obtained.

Determine the moisture in the hide powder by drying to constant weight at 98° to 100° C. and calculate the amount of tenth-normal alkali required to neutralize 10 gm. of dry hide powder. From 12 to 13 mls should be required.

* Journal American Leather Chemists' Association, 11, 549 (1916).

HYDRAZINE SULPHATE**(Diamine Sulphate)** $(\text{NH}_2)_2\text{H}_2\text{SO}_4$

Mol. Wt. 130.13

Hydrazine sulphate occurs usually as colorless crystals or a white crystalline powder, soluble in about 40 parts of water but insoluble in alcohol. Its theoretical melting point is 254°C . The aqueous solution is acid to litmus. The reagent contains at least 99% of $(\text{NH}_2)_2\text{H}_2\text{SO}_4$.

Hydrazine sulphate is used principally in the determination of copper, in the detection of blood, and in the separation and determination of arsenic.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Heavy Metals	0.0000%
Non-volatile Matter	0.0500%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 50 mls of water and add nitric acid and silver nitrate solution. Not more than a slight opalescence should be produced.

Heavy Metals.—Dissolve 1 gm. in 50 mls of water and add hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia and a few drops of ammonium sulphide solution. No dark color or precipitate should result.

Non-volatile Matter.—Ignite 2 gm. of hydrazine sulphate and weigh the residue. The weight should not exceed 0.001 gm.

Quantitative Method.—Dissolve about 0.1 gm., accurately weighed, in 20 mls of water, add 1 gm. of sodium bicarbonate and titrate with tenth-normal iodine solution, using starch solution as indicator. At least 99% of $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ should be found.

1 mil of tenth-normal I = 0.00325325 gm. of $(\text{NH}_2)_2\text{H}_2\text{SO}_4$,
log 51231.

HYDROGEN SULPHIDE WATER

Hydrogen sulphide water is a clear, colorless liquid, having a strong odor of hydrogen sulphide. It has an acid reaction to litmus paper. When freshly prepared the reagent consists of a saturated solution of hydrogen sulphide gas in water, but the gas is gradually lost on exposure to the air by volatilization and also by decomposition with deposition of sulphur. Light favors the oxidizing action of the air.

At ordinary temperature water absorbs about 3 volumes of hydrogen sulphide gas. Ether and alcohol are capable of absorbing much more. In ether solution the gas is quite stable but alcoholic solutions quickly decompose.

Hydrogen sulphide water is used as a precipitant of the heavy metals. It should be kept in tightly-stoppered bottles in a cool place and protected from light. When intended to be stored and used over a period of time it is best kept in small, completely filled bottles.

Maximum Limit of Impurity

Iron 0.0000%

Method of Testing

Iron.—Hydrogen sulphide water should not be changed in appearance upon the addition of ammonia water.

HYDROXYLAMINE HYDROCHLORIDE

$\text{NH}_2\text{OH}.\text{HCl}$

Mol. Wt. 69.50

Hydroxylamine hydrochloride usually occurs as colorless, hygroscopic crystals, soluble in about 1 part of water and in about 15 parts of alcohol; it is also soluble in glycerin. The reagent contains at least 95% of $\text{NH}_2\text{OH}.\text{HCl}$. Its solutions are acid to litmus.

Hydroxylamine is a strong reducing agent. It precipitates gold, silver and mercury in metallic form from their solutions, decolorizes permanganate in acid or neutral solution, and reduces Fehling's solution, even in the cold. As

a reagent it is used principally in the determination of gold, silver and glucose; with benzidine in determining sulphates in water; in testing for mercury, copper, acetone, acetylene and colchicine; and in organic analysis and synthesis.

The salt should be stored in well-closed, glass-stoppered bottles. It should not be exposed to moist air, since it is hygroscopic and in moist condition decomposes.

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Ammonium Chloride (NH_4Cl)	0.3000%
Sulphates (SO_4)	0.0015%
Heavy Metals	Trace
Arsenic (As)	0.0015%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the salt. Not more than 0.001 gm. of residue should remain.

Ammonium Chloride.—Dissolve 1 gm. in 20 mls of alcohol and add platinic chloride solution. No precipitate should form.

Sulphates.—Dissolve 2 gm. in 20 mls of water, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. Not more than a slight coloration should result.

Arsenic.—Mix 1 gm. with 3 mls of stannous chloride solution and allow it to stand one hour. No dark color should develop.

Quantitative Method.—Dissolve about 0.1 gm., accurately weighed, in 10 mls of water, add 20 mls of a cold, saturated solution of ferric ammonium sulphate and 15 mls of 10% sulphuric acid, boil the mixture five minutes, dilute it with 25 mls of boiled water and titrate with tenth-

normal potassium permanganate solution. At least 95% of $\text{NH}_2\text{OH.HCl}$ should be found.

1 mil of tenth-normal $\text{KMnO}_4 = 0.003475$ gm. of $\text{NH}_2\text{OH.HCl}$, log 54095.

INDIGO

(Synthetic Indigo)

Indigo for reagent use is usually in the form of a fine, dark blue powder. It is insoluble in water or in alcohol. It contains at least 95% of $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_2$ (indigotin). Commercial indigo mixed with water, thus forming a paste, is also on the market. The dehydrated form, however, is the kind usually employed as a reagent.

Indigo, on treatment with fuming sulphuric acid, is converted to a disulphonic acid which is soluble in water and in the form of its sodium salt is known as indigo carmine. (See Sodium Indigotinsulphonate, page 326.) Indigo is decolorized by reducing substances, such as bisulphites, glucose, oil of turpentine, etc., the blue color returning upon exposure to air for some time. Chlorine and nitric acid also decolorize indigo and upon this behavior is based the detection and determination of nitric acid which constitute the principal use of the reagent. For qualitative purposes a 1:1000 solution is usually employed (see page ix).

Solutions of indigo do not keep indefinitely and when they acquire a greenish or brownish tinge should be rejected.

Maximum Limits of Impurities

Ash	1.0000%
Moisture	1.0000%

Methods of Testing

Ash.—Ignite 0.5 gm. and weigh the ash. The weight should not exceed 0.005 gm.

Moisture.—Dry the indigo to constant weight at 100°C . The loss in weight should not exceed 1%.

Quantitative Method.—Dry the indigo at 100° C. to constant weight, weigh out 0.5 gm. of the dried sample, mix it in a beaker with 10 mls of sulphuric acid, warm the mixture on the steam-bath, stirring frequently until solution is complete and dilute it to 1 liter. To 50 mls of the solution add 600 mls of water and titrate with tenth-normal potassium permanganate solution. The end-point is the change from green to light yellow, not the incipient red color. At least 95% of $C_{16}H_{10}N_2O_2$ (indigotin) should be found.

1 mil of tenth-normal $KMnO_4 = 0.007493$ gm. of $C_{16}H_{10}N_2O_2$, log 87466.

Note: Regarding this titration see J. Chem. Soc., 94, I, 468 (1908).

IODEOSIN

(Tetraiodofluorescein)

$C_{20}H_8I_4O_5$

Mol. Wt. 835.84

Iodeosin is a scarlet-red powder. It is insoluble in water containing a trace of acid, but is readily soluble in alcohol, forming a deep red solution, and in ether, yielding a yellowish-red solution.

Iodeosin solution to be used as an indicator is made by dissolving 0.1 gm. in 100 mls of alcohol. About 6 drops of this solution are sufficient for 50 to 100 mls of the liquid to be titrated. After adding the iodeosin, a layer of about 30 mls of ether is superimposed upon the aqueous liquid. On shaking, this ether layer takes up and holds the indicator if the aqueous portion contains even a slight excess of acid, while a slight excess of alkali again abstracts the indicator from the ethereal layer, producing a pink color in the aqueous portion.

Iodeosin is especially adapted to the estimation of small amounts of acids and alkalis by the use of the more dilute volumetric solutions, such as hundredth-normal. It is used largely in the titration of alkaloids. The mono-sodium salt of diethylbarbituric acid may be titrated with it.

Method of Testing

Sensitiveness.—Into a well rinsed, colorless, glass-stoppered flask place 100 mls of water and add 5 drops of the above described iodeosin indicator solution and 30 mls of ether. Then add drop by drop hundredth-normal hydrochloric acid solution, shaking the mixture thoroughly after each addition, until the aqueous layer just becomes colorless. Now add 5 drops more of the iodeosin solution and shake again. The aqueous layer should not acquire a pink color, or, should it do so, the addition of 1 drop of the hundredth-normal acid should again render it colorless.

IODINE

I**At. Wt. 126.92**

Iodine usually occurs as dry, heavy, bluish-black scales or plates having a metallic luster. It is readily soluble in alcohol (about 10 parts), ether, chloroform and carbon disulphide (about 5 parts); only slightly soluble in water (about 3,000 parts), but easily soluble in solutions of the alkaline iodides. It contains at least 99.98% of I.

Iodine melts when heated to about 114° C. and volatilizes at ordinary temperatures. Aqueous solutions of iodine gradually lose strength on keeping, due to volatilization of the iodine and to the action of air and light. Volumetric solutions should therefore be restandardized frequently.

Iodine has a wide range of usefulness as a reagent, particularly in volumetric analysis in which it serves in the determination of sulphites, sulphides, thiosulphates, arsenic, antimony, mercury, tin, acetone, etc. It is used in organic synthesis and microscopy; in the determination of the iodine number of fats and oils; in the detection of starch, cellulose, tannic and gallic acids, bismuth, etc.; and as an alkaloidal reagent.

Iodine should be kept in well-closed, glass-stoppered containers in a cool place. Its solutions should be stored away from the light.

Maximum Limits of Impurities

Non-volatile Matter	0.0100%
Cyanogen Compounds (CN)	0.0050%
Chlorine and Bromine (Cl)	0.0120%

Methods of Testing

Non-volatile Matter.—Cautiously heat 10 gm. in a porcelain dish until the iodine is completely volatilized. The weight of the residue should not exceed 0.001 gm.

Cyanogen, Chlorine and Bromine.—Powder 5 gm. of iodine, shake it thoroughly with 25 mil of water and, after it has stood for half an hour, filter and divide the filtrate into two equal portions. To one add sulphurous acid, drop by drop, until decolorized, then add a few milligrams of ferrous sulphate, a drop of ferric chloride solution and 2 to 3 mls of 5% sodium hydroxide solution and heat the mixture to about 60° C. Then add a slight excess of hydrochloric acid. The liquid should not acquire a blue color.

To the other portion of the filtrate add 1 mil of ammonia water and 0.25 mil of tenth-normal silver nitrate solution; filter and to the clear filtrate add 2 mls of nitric acid. The liquid should not become more than opalescent.

Quantitative Method.—Dissolve about 0.5 gm. of iodine, accurately weighed, with 1 gm. of potassium iodide in 5 mls of water, dilute to about 50 mls and titrate with tenth-normal sodium thiosulphate solution. At least 99.98% of I should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.012692$ gm. of I,
log 10353.

IODINE WATER

Iodine water is a saturated, or nearly saturated, aqueous solution of iodine. It contains at least 0.2 gm. of I per liter.

The solution under the influence of air and light loses its iodine content, hydriodic acid being gradually formed. The

use of amber bottles is said not to retard the change materially, but a large air space in the containers appreciably accelerates the decomposition. With the acquisition of hydriodic acid the solution becomes capable of dissolving more iodine than will dissolve in pure water, but the presence of the acid in appreciable amounts in the reagent is generally undesirable.

Iodine water is used principally as a reagent in microscopy and for detecting starch, narceine, cocaine and other alkaloids. It should be stored in completely filled, glass-stoppered containers in a dark place.

Method of Testing

Quantitative Method.—Titrate the iodine water with a standard solution of sodium thiosulphate. At least 0.2 gm. of I per liter should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.012692$ gm. of I,
log 10353.

IRON WIRE

(Piano Wire)

Fe

At. Wt. 55.84

Iron wire is a fine, bright wire usually obtained wound upon spools or cut into lengths of convenient size. It is soluble in dilute hydrochloric or sulphuric acid, evolving practically odorless hydrogen gas and leaving not more than traces of undissolved black residue.

Iron wire is used principally for standardizing solutions of potassium permanganate. Its iron value, however, is not dependable on account of the invariable presence of carbon which, upon dissolving the iron in acids, is converted into hydrocarbons exerting a strong reducing action upon permanganate. On this account iron wire has been superseded to considerable extent by sodium oxalate. Nevertheless it is a convenient and unalterable substance for the purpose, provided its effective value has been accurately de-

terminated against sodium oxalate or other reliable standard. Regarding the method of obtaining the titrimetric value of the iron wire and the precautions to be observed see Treadwell-Hall's *Analytical Chemistry*, 4th ed., II, 98 and 601 (1915).

Besides the above-mentioned use, iron wire is employed as a reducing agent, in the detection of copper and antimony and in preparing empirical solutions of iron chloride used for colorimetric and other determinations.

The reagent should be protected from moist air, acid fumes, etc., by storage in well-closed, glass containers.

IRON BY HYDROGEN

(Reduced Iron)

Fe

At. Wt. 55.84

Iron by hydrogen is a fine, gray powder, insoluble in water, soluble in hydrochloric or dilute sulphuric acid with evolution of hydrogen and the formation of an iron salt. It contains at least 90% of metallic Fe.

Iron by hydrogen is mainly used in connection with zinc dust for the detection and determination of nitrates by reduction to ammonia.

Maximum Limits of Impurities

Substances Insoluble in Sulphuric Acid	0.5000%
Water-soluble Matter	0.0600%
Nitrogen (N)	0.0028%
Arsenic (As)	0.0015%

Methods of Testing

Substances Insoluble in Sulphuric Acid.—Dissolve 1 gm. in 25 mls of diluted sulphuric acid by warming on a steam-bath until evolution of gas has ceased and collect and weigh any undissolved residue. The weight should not exceed 0.005 gm.

Water-soluble Matter.—Shake 5 gm. with 50 mls of water, filter, evaporate the filtrate on the steam-bath and weigh the residue. The weight should not exceed 0.003 gm.

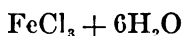
Nitrogen.—Dissolve 20 gm. in a mixture of 20 mls of sulphuric acid and 200 mls of water with the aid of heat. When the solution has cooled add 100 mls of 27% solution of sodium hydroxide and distil off about 75 mls, collecting the distillate in a receiver containing 10 mls of tenth-normal solution of hydrochloric acid. Titrate the excess acid with tenth-normal sodium hydroxide solution, using methyl orange as indicator. The ammonia from the iron should not neutralize more than 0.4 ml of the tenth-normal acid solution.

Arsenic.—Pour 10 mls of hydrochloric acid over a mixture of 1 gm. of the iron and 1 gm. of potassium chlorate and, when the reaction has subsided, heat until all free chlorine has been expelled. Filter, add to the filtrate 15 mls of stannous chloride solution and allow to stand one hour. A dark color should not develop.

Quantitative Method.—Place in a 100-ml flask about 1 gm. of the iron, accurately weighed, add 10 gm. of powdered mercuric chloride and 50 mls of boiling water and boil the mixture for about 5 minutes with frequent shaking. Make up the volume with water, recently boiled and cooled, until it measures 100 mls at room temperature, shake thoroughly, filter and at once titrate an aliquot with tenth-normal solution of potassium permanganate. At least 90% of Fe should be found.

1 ml of tenth-normal KMnO_4 = 0.005584 gm. of Fe,
log 74695.

IRON CHLORIDE, FERRIC



Mol. Wt. 270.32

Ferric chloride usually occurs as brownish-yellow, very deliquescent, crystalline masses, soluble in less than 1 part of water and also readily soluble in alcohol, yielding solutions acid to litmus. The salt contains at least 95% of $\text{FeCl}_3 + 6\text{H}_2\text{O}$.

When heated to about 37° C. ferric chloride melts to a reddish-brown liquid. At higher temperatures the salt loses water and hydrochloric acid and acquires a content of insoluble basic salt. The reagent usually contains a little free hydrochloric acid which insures the absence of basic salt.

Ferric chloride is principally used in the detection of acetic, salicylic and tannic acids, cyanides, ferrocyanides and sulphocyanides; for detecting acetoacetic acid and indican in urine; as a reagent for pyrocatechin, antipyrine, santonin, phenol, iodides and alkaloids; in the volumetric determination of tin and fluorine; and in the decomposition of earthy phosphates.

The reagent should be stored in tightly-stoppered bottles in a cool place, and should be protected from direct sunlight.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Free Chlorine (Cl)	0.0010%
Arsenic (As)	0.0023%
Ferrous Salt (Fe'')	0.0025%
Zinc (Zn)	0.0015%
Copper (Cu)	0.0050%
Calcium and Alkalis	0.0240%
Nitric Acid (HNO ₃)	0.0250%
Sulphates (SO ₄)	0.0018%

Methods of Testing

Insoluble Matter (Basic Salt).—Dissolve 10 gm. of the salt in 10 mils of water. A perfectly clear solution should result.

Free Chlorine.—Boil 10 mils of the solution obtained in the preceding test and test the vapors with zinc iodide-starch paper. The paper should not turn blue.

Arsenic.—Mix 1 mil of the solution obtained in the test for basic salt with 3 mils of stannous chloride solution and allow it to stand one hour. The mixture should not acquire a darker color in this time.

Ferrous Salt.—Dissolve 1 gm. in 20 mls of water and add 1 mil of hydrochloric acid and a few drops of potassium ferricyanide solution. No blue color should result.

Zinc, Copper, Calcium, Alkalis and Nitric Acid.—Dilute 20 mls of a 50% solution of the ferric chloride with 100 mls of water, add 30 mls of ammonia water, filter and bring the filtrate to a volume of 75 mls by washing. Evaporate 50 mls of the filtrate and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Mix 2 mls of the filtrate with 2 mls of sulphuric acid and overlay the mixture with 1 mil of ferrous sulphate solution. No brown color should develop at the plane of contact of the liquids.

Acidulate the remainder of the filtrate with acetic acid, add potassium ferrocyanide solution and allow it to stand two hours. No white or brownish-red precipitate should form.

Sulphates.—Dissolve 10 gm. of ferric chloride in 100 mls of water, add 25 mls of ammonia water, filter, acidulate the filtrate with acetic acid, add barium chloride solution and allow it to stand twelve to eighteen hours. Any precipitate of barium sulphate forming should not weigh more than 0.0005 gm.

Quantitative Method.—Dissolve about 1 gm., accurately weighed, in 100 mls of water, add 2 gm. of potassium iodide and 5 mls of hydrochloric acid, allow the mixture to stand half an hour in a stoppered flask at about 40° C., cool it and titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 95% of $\text{FeCl}_3 + 6\text{H}_2\text{O}$ should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.027032$ gm. of
 $\text{FeCl}_3 + 6\text{H}_2\text{O}$, log 43188.

IRON CHLORIDE, FERRIC, SOLUTION

Iron chloride, ferric, solution, is a clear, yellowish-brown liquid having a specific gravity of about 1.280. It con-

tains from 9.75% to 10.25% of Fe, equivalent to about 29% of FeCl_3 .

This solution offers a convenient form of ferric chloride for the ordinary stock solution. It is not subject to changes which might render the crystallized salt unfit for use, such as those due to heat or absorption of water. It represents approximately 50% of the reagent described under Iron Chloride, Ferric, and the amount of the solution to be used instead of that article can therefore readily be calculated.

The solution should be stored in glass-stoppered bottles, protected from light. Ammonia fumes, often present in the air of laboratories, will gradually cause precipitation in the solution if allowed to come in contact with it frequently or continuously.

Maximum Limits of Impurities

Free Chlorine (Cl)	0.0005%
Arsenic (As)	0.0012%
Ferrous Salt (Fe^{+2})	0.0032%
Zinc (Zn)	0.0008%
Copper (Cu)	0.0025%
Calcium and Alkalis	0.0117%
Nitric Acid (HNO_3)	0.0125%
Sulphates (SO_4)	0.0012%

Methods of Testing

Free Chlorine.—Boil 10 mils of the solution and test the vapors with zinc iodide-starch paper. The paper should not turn blue.

Arsenic.—Mix 1 mil with 3 mils of stannous chloride solution and allow it to stand one hour. The mixture should not acquire a darker color in this time.

Ferrous Salt.—Dilute 1 mil with 20 mils of water and add 1 mil of hydrochloric acid and a few drops of potassium ferricyanide solution. No blue color should result.

Zinc, Copper, Calcium, Alkalis and Nitric Acid.—To 20 mils of the solution apply the tests as directed under Iron Chloride, Ferric. The results should be as there stated.

Sulphates.—Mix 15 mls of the solution with 100 mls of water, add 25 mls of ammonia water, filter, acidulate the filtrate with acetic acid, add barium chloride solution and allow it to stand twelve to eighteen hours. Any precipitate forming should not weigh more than 0.0005 gm.

Quantitative Method.—Accurately weigh about 2 gm. of the solution, add 25 mls of water, 5 mls of hydrochloric acid and 3 gm. of potassium iodide, and allow the mixture to stand half an hour in a stoppered flask at about 40° C. Cool, dilute with 100 mls of water and titrate the liberated iodine with tenth-normal sodium thiosulphate solution, using starch solution as indicator. From 9.75% to 10.25% of Fe should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.005584$ gm. of Fe,
log 74695.

IRON CHLORIDE, FERROUS

$\text{FeCl}_2 + 4\text{H}_2\text{O}$

Mol. Wt. 198.82

Iron chloride, ferrous, is usually in the form of deliquescent, pale green crystals or powder, soluble in about 1 part of water acidulated with hydrochloric acid; also soluble in alcohol. On exposure to air it easily oxidizes with the formation of basic salt, small amounts of which are always present. The addition of hydrochloric acid is therefore always necessary to effect complete solution of the salt. Extensive oxidation is recognized by the appearance of yellow spots on the crystals and a yellow color in the solution.

The reagent is used principally as a reducing agent in inorganic analyses; in the gasometric determination of nitrates; and in the determination of nitric acid.

Ferrous chloride should be stored in well-stoppered bottles and should be exposed to the air as little as possible.

Maximum Limits of Impurities

Oxychloride	Small Amount
Sulphates (SO_4)	0.0075%
Copper (Cu)	0.0300%

Zinc (Zn)	0.0075%
Salts of the Alkalis	0.0900%
Arsenic (As)	0.0015%

Methods of Testing

Oxychloride.—Dissolve 1 gm. in 1 mil of water acidulated with 2 or 3 drops of hydrochloric acid. The solution should be pale green without a yellowish tint. Add hydrogen sulphide to the solution. Only a slight turbidity due to separation of sulphur should result.

Sulphates, Copper, Zinc and Salts of the Alkalis.—Dissolve 5 gm. in 10 mils of water, add 5 mils of nitric acid and heat until brown in color. Dilute to 120 mils, add 30 mils of ammonia water, filter and bring the colorless filtrate to a volume of 75 mils by washing.

Evaporate 50 mils of the filtrate to dryness and ignite and weigh the residue. The weight should not exceed 0.003 gm.

Acidulate 20 mils of the filtrate with acetic acid, add barium chloride solution and allow to stand five minutes. The liquid should not become cloudy.

Acidify the remainder of the filtrate with acetic acid, add potassium ferrocyanide solution and allow it to stand two hours. No white or brownish-red precipitate should form.

Arsenic.—Dissolve 1 gm. in 1 mil of water acidulated with a few drops of hydrochloric acid, add 3 mils of stannous chloride solution and allow to stand one hour. The mixture should not acquire a darker color.

IRON SULPHATE, FERROUS

$\text{FeSO}_4 + 7\text{H}_2\text{O}$

Mol. Wt. 278.01

Ferrous sulphate is usually obtained as pale bluish-green crystals or granules, odorless, and having a saline, styptic taste. The salt is efflorescent in dry air. It is soluble in less than 2 parts of water but insoluble in alcohol. The salt when exposed to the air gradually oxidizes, the presence of

basic ferric salt being indicated by the formation of yellow spots on the crystals and imperfect solubility. The crystals of ferrous sulphate should be clear and should have a distinct bluish tinge; a grass-green color would denote incipient oxidation.

Ferrous sulphate is used principally as a reducing agent, serving in this capacity in the determination of gold, manganese dioxide, and in many other operations. It is also used in the detection of nitric acid, cyanides and ferrocyanides, tannic acid, benzoic acid, vanillin, ethyl peroxide, etc.

The reagent should be kept in well-stoppered bottles. Its solutions readily deteriorate and should be freshly prepared when required.

Maximum Limits of Impurities

Insoluble Substances	0.0000%
Salts of the Alkalis	0.0300%
Copper (Cu)	0.0120%
Zinc (Zn)	0.0036%
Phosphorus Compounds (P)	0.0010%

Methods of Testing

Insoluble Matter (Basic Iron Sulphate).—Dissolve 1 gm. in 20 mils of water which has been thoroughly boiled and then cooled. The solution should be clear and have a greenish-blue color.

Salts of the Alkalis.—Dissolve 5 gm. of ferrous sulphate in 100 mils of water, add 10 mils of nitric acid and boil for several minutes; then add 25 mils of ammonia water, filter, and bring the filtrate to a volume of 75 mils by washing. The filtrate should be colorless. Evaporate 50 mils of the filtrate and ignite and weigh the residue. The weight should not exceed 0.001 gm.

Copper and Zinc.—Add a slight excess of acetic acid to 25 mils of the filtrate obtained in the preceding test, then add potassium ferrocyanide solution and allow it to stand two hours. No white or brownish-red precipitate should form.

Phosphorus Compounds.—Dissolve 10 gm. of the ferrous sulphate in 100 mls of water, add 20 mls of nitric acid, sp. gr. 1.40–1.42, and heat the solution on a steam-bath for half an hour. Then add 50 mls of ammonium molybdate solution and allow the mixture to stand at about 40° C. for eighteen to twenty-four hours. No yellow precipitate should form in this time.

Quantitative Method.—Dissolve about 1 gm. of the ferrous sulphate, accurately weighed, in 50 mls of well-boiled water, add 10 mls of sulphuric acid and titrate with tenth-normal solution of potassium permanganate.

1 mil of tenth-normal $\text{KMnO}_4 = 0.027801$ gm. of $\text{FeSO}_4 + 7\text{H}_2\text{O}$, log 44406.

IRON SULPHIDE

(Ferrous Sulphide)

FeS

Mol. Wt. 87.9

Ferrous sulphide for laboratory use is obtainable in different forms, such as lumps, sticks, granules, globules, etc., of a dark gray or grayish-black color. It is soluble in diluted sulphuric or hydrochloric acid with the copious evolution of hydrogen sulphide. It contains at least 65% of FeS .

A method of determining hydrogen sulphide obtained in the decomposition of ferrous sulphide has been proposed [H. Williams, Chem. News, 116, 13 (1917)] in which the gas is passed into cadmium chloride solution, the precipitated cadmium sulphide redissolved and the cadmium determined by electrolysis, from the result of which the amount of hydrogen sulphide is deduced. Regarding other methods involving the use of cadmium salts see Scott's Standard Methods of Chemical Analysis, 2d ed., 407 (1918).

Iron sulphide is employed for the production of hydrogen sulphide.

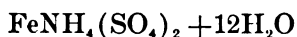
Method of Testing

Quantitative Method.—Place 0.4 to 0.5 gm. of the finely powdered ferrous sulphide, accurately weighed, in a distillation flask, and after the outlet tube has been connected with a receiver, or better a series of two or more receivers, containing altogether about 100 mls of tenth-normal iodine solution, introduce through a funnel-tube having a stop-cock 50 mls of 10% sulphuric acid, and heat the contents of the flask to boiling until the hydrogen sulphide has been entirely driven off. Titrate the iodine solutions, which should not have been entirely decolorized during the distillation, with tenth-normal sodium thiosulphate solution. At least 65% of FeS should be found.

1 mil of tenth-normal I = 0.004395 gm. of FeS, log 64296.

IRON AND AMMONIUM SULPHATE, FERRIC

(Ferric Ammonium Sulphate; Ammonio-Ferric Alum)



Mol. Wt. 482.19

Ferric ammonium sulphate is generally in the form of pale violet, transparent crystals, soluble in about 3 parts of water but insoluble in alcohol. The salt is efflorescent and odorless and has an acid, styptic taste and an acid reaction. It usually contains 99% to 100% of $(\text{FeNH}_4(\text{SO}_4)_2 + 12\text{H}_2\text{O})$.

This salt finds its principal use as an indicator in the titration of halogens by means of silver nitrate and ammonium sulphocyanate volumetric solutions. A few mls of the 10% solution are generally sufficient for the purpose. Ferric ammonium sulphate has also been used to some extent for standardizing permanganate solutions, the iron being first reduced to the ferrous condition by stannous chloride. For this purpose the exact iron content of the salt must first be established by gravimetric or volumetric means.

The reagent should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Ferrous Salt (Fe ²⁺)	0.0025%
Chlorides (Cl)	0.0010%
Zinc (Zn)	0.0030%
Copper (Cu)	0.0100%
Salts of the Alkalis	0.0400%

Methods of Testing

Ferrous Salt.—Dissolve 1 gm. in 20 mls of water and add 1 mil of hydrochloric acid and 1 drop of freshly prepared solution of potassium ferricyanide. No blue or green color should develop.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 2 or 3 mls of nitric acid and some silver nitrate solution. No change should be apparent.

Zinc and Copper.—Dissolve 2 gm. in 50 mls of water, add 10 mls of ammonia water and filter. The filtrate should be colorless. Acidulate with acetic acid, add potassium ferrocyanide solution and allow it to stand two hours. No white or brownish-red precipitate should form.

Salts of the Alkalis.—Dissolve 5 gm. of ferric ammonium sulphate in 100 mls of water, add 15 mls of ammonia water, filter, evaporate the filtrate and ignite and weigh the residue. The weight of the residue should not exceed 0.002 gm.

Quantitative Method.—Dissolve about 1 gm. of the salt, accurately weighed, in 100 mls of water, and then add 2 gm. of potassium iodide followed by 5 mls of hydrochloric acid. Allow the mixture to stand, well stoppered, for half an hour and titrate the iodine liberated by the reaction with tenth-normal solution of sodium thiosulphate.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.0428194$ gm. of
 $\text{FeNH}_4(\text{SO}_4)_2 + 12\text{H}_2\text{O}$, log 68322.

IRON AND AMMONIUM SULPHATE, FERROUS (Ferrous Ammonium Sulphate)



Mol. Wt. 392.14

Ferrous ammonium sulphate is usually obtained as pale bluish-green crystals or granules, soluble in about 6 parts of water; insoluble in alcohol. It contains generally 99% to 100% of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 + 6\text{H}_2\text{O}$.

This salt is much more stable than ferrous sulphate and under good conditions of storage will keep unchanged for a long time. On this account it is often substituted for the latter for various reagent purposes, particularly in volumetric analysis, its solutions undergoing only very gradual change. Hence it is employed in the determination of manganese dioxide and chromic acid and of vanadium in steel. The salt carefully prepared to avoid excess moisture and to insure proper composition is sometimes used in standardizing dichromate and permanganate solutions, having some advantage over metallic iron; but sodium oxalate has become the generally preferred starting material for volumetric permanganate solutions. Directions for making a pure ferrous ammonium sulphate in the laboratory for use as a volumetric standard are given in Sutton's Volumetric Analysis, 10th ed., 123 (1911).

The reagent should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Ferric Salts (Fe''')	0.0008%
Copper (Cu)	0.0100%
Zinc (Zn)	0.0030%
Alkali Salts	0.0167%

Methods of Testing

Ferric Salts.—Dissolve 1 gm. of ferrous ammonium sulphate in 20 mls of thoroughly boiled water, add 1 mil of hydrochloric acid and some potassium sulphocyanate solution. No red color should appear at once.

Copper and Zinc.—Dissolve 5 gm. in 50 mls of water, add 10 mls of nitric acid and boil the solution for a few minutes. Add 15 mls of ammonia water, filter, and bring the filtrate to a volume of 50 mls by washing. The filtrate should be colorless. To 20 mls add a slight excess of acetic acid and then some potassium ferrocyanide solution. No white or brownish-red precipitate should form.

Alkali Salts.—Evaporate 30 mls of the filtrate obtained in the test for copper and zinc, and ignite and weigh any residue remaining. The weight should not exceed 0.0005 gm.

Quantitative Method.—Dissolve the salt in well-boiled and cooled water, add several mls of diluted sulphuric acid and titrate with tenth-normal potassium permanganate solution.

1 ml of tenth-normal $\text{KMnO}_4 = 0.039214$ gm. of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 + 6\text{H}_2\text{O}$, log 59344.

ISATIN

$\text{C}_8\text{H}_5\text{NO}_2$

Mol. Wt. 147.12

Isatin is an oxidation product of indigo and occurs usually as small, yellowish-red crystals, slightly soluble in cold water but readily soluble in hot water, alcohol or ether. It melts at 200° to 201° C. and at higher temperatures sublimes. Solutions of isatin in water, alcohol or ether are reddish-brown. It dissolves in alkali hydroxide solutions yielding violet-colored solutions which on standing or on heating become yellow.

Isatin is used chiefly for the detection of thiophene and mercaptans and in the determination of indican in urine.

LACMOID

(Resorcinol Blue)

Lacmoid usually occurs as lustrous, dark violet scales or granules, soluble in alcohol and acetone, less soluble in water and in ether.

The constitution of lacmoid is not at present known with certainty, but the formula, $C_{12}H_6NO_8$, has been assigned to it. A substance termed "lackmusol," which is stated to be the sensitive constituent of lacmoid, has been described and its method of preparation given (see Chem. Abst., 8, 3542 [1914]).

When lacmoid is boiled with water it should color the water an intense blue. An inferior and less soluble preparation can be distinguished by its failure to do this. The alcoholic solution should have a pure blue color without a violet tinge. To make an indicator solution dissolve 0.2 gm. in 100 mls of alcohol. About 0.2 mil of this solution may be added to a volume of 100 mls of the liquid to be titrated.

Lacmoid is useful in the titration of mineral acids and strong bases, such as ammonia and hydroxides of the alkalis and alkali earths; also in the titration of various alkalis. It is unsuited to the determination of organic acids. Carbonates may be determined with it by residual titration after boiling off the carbon dioxide. It is employed in water analysis for determining the alkalinity and temporary hardness of waters. The endpoint is generally sharper in cold than in hot solutions.

Solutions of lacmoid should be protected from light, exposure to which soon causes them to deteriorate.

Method of Testing

Sensitiveness.—Add 0.2 mil of the alcoholic solution of lacmoid, prepared as above directed, to 100 mls of water which has been freed from carbon dioxide by long boiling in a platinum dish. Add 0.05 mil of tenth-normal hydrochloric acid solution to the liquid. The blue color should change to red, and should be restored by the subsequent addition of 0.05 mil of tenth-normal sodium hydroxide solution.

LEAD, FREE FROM SILVER**(Test Lead)****Pb**

At. Wt. 207.2

Lead, free from silver, is usually obtainable in the form of granules, sheets or sticks, bluish-gray in color and having a dull metallic luster. It is readily soluble in nitric acid.

The reagent is employed chiefly in the dry assay of ores, especially those containing gold and silver; and as a reducing agent, particularly for iron previous to its oxidimetric titration.

Maximum Limits of Impurities

Silver (Ag)	0.0010%
Arsenic (As)	0.0002%
Other Foreign Heavy Metals	Trace
Foreign Non-volatile Matter	0.0100%

Methods of Testing

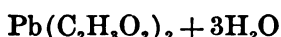
Silver.—Dissolve 10 gm. of the lead in 25 mls of nitric acid, add 75 mls of 10% sulphuric acid and evaporate the mixture, first on the steam-bath and later on a sand-bath or hot-plate, with frequent stirring until the nitric acid has been expelled and fumes of sulphuric acid begin to come off. Mix the mass with 50 mls of water, add 50 mls of alcohol, mix well and allow to stand until cold. Filter, evaporate the filtrate to about 30 mls in order to expel the alcohol, cool it and add a few drops of dilute hydrochloric acid. No change should be apparent. Dilute the liquid to a volume of 40 mls for further testing.

Arsenic.—Introduce 10 mls of the solution remaining in the preceding test into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

Other Foreign Heavy Metals.—To 10 mls of the solution remaining in the test for silver add a slight excess of ammonia water and a few drops of ammonium sulphide solution. A brownish color may develop but no precipitate should form.

Foreign Non-volatile Matter.—Evaporate 20 mls of the solution remaining in the test for silver, and ignite and weigh the residue. The weight should not exceed 0.005 gm.

LEAD ACETATE



Mol. Wt. 379.32

Lead acetate is usually in the form of colorless crystals, easily soluble in about 2 parts of water. It slowly effloresces and also gradually takes up carbon dioxide on exposure to air and then it exhibits a turbidity in its solutions. Even the carbon dioxide ordinarily dissolved in water often interferes with the transparency of lead acetate solutions, so that water freed from carbon dioxide by boiling should be used in testing the solubility of the salt or in making a clear, aqueous solution.

Lead acetate is used in the determination of chromic and molybdic acids and as a precipitant for tannic, malic and oxalic acids. It is a constituent of reagents for dextrose, cottonseed oil, dextrin and indican and is also used for testing liquid petrolatum for sulphur compounds. Lead acetate paper is a sensitive reagent for hydrogen sulphide gas, detecting even 1 part in 200,000.

Lead acetate should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Earths and Alkalis	0.0500%
Copper (Cu)	0.0010%
Iron (Fe)	0.0020%
Insoluble Matter	Trace
Chlorides (Cl)	0.0010%
Nitrates (N_2O_5)	0.0032%

Methods of Testing

Earths and Alkalis.—Dissolve 10 gm. of lead acetate in about 200 mls of water and precipitate the lead from the solution with hydrogen sulphide gas. Filter, evaporate the filtrate and ignite the residue. Its weight should not exceed 0.005 gm.

Copper and Iron.—Dissolve 2 gm. of lead acetate in 10 mls of water, mix the solution with 3 mls of sulphuric acid, filter and to the filtrate add an excess of ammonia water. No precipitate or blue color should be produced.

Insoluble Matter (Carbonate, Silicate, etc.).—Dissolve 5 gm. in 50 mls of freshly boiled water. The solution should be clear or at most very slightly opalescent.

Chlorides.—Dissolve 1 gm. in 30 mls of water and add a little nitric acid and silver nitrate solution. The solution should appear unchanged.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add 1 drop of indigo solution and 10 mls of sulphuric acid and mix thoroughly. The blue color should not be entirely discharged.

LEAD CHROMATE

PbCrO_4

Mol. Wt. 323.0

Lead chromate is usually in the form of brown, fused lumps or a yellowish-brown powder, insoluble in water, but almost completely soluble in solutions of the fixed alkali hydroxides and also, with decomposition, in nitric acid.

Upon heating, the lead chromate melts to a brown mass without decomposition. At a red heat the salt completely oxidizes all organic substances and on this account is employed in elementary analysis to assist combustion, especially of organic bodies containing halogens, sulphur, or phosphorus. Before use for this purpose the reagent should be carefully ignited in a current of oxygen (air is less efficient) to remove all traces of carbon dioxide or organic matter.

Lead chromate should be stored in well-stoppered bottles and protected from contamination with dust or other organic matter.

Maximum Limit of Impurity

Substances Soluble in Water 0.1000%

Method of Testing

Substances Soluble in Water.—Powder the lead chromate and shake 5 gm. with 50 mls of water, warmed to about 50° C., for five minutes. Filter, evaporate the filtrate to dryness, ignite the residue and weigh it. The weight should not exceed 0.005 gm.

LEAD NITRATE

$\text{Pb}(\text{NO}_3)_2$

Mol. Wt. 331.22

Lead nitrate usually occurs as white, translucent crystals or a crystalline powder, soluble in about 2 parts of water, almost insoluble in alcohol. It is less soluble in water containing nitric acid than in water alone.

The reagent is used principally for detecting sulphides; for preparing solutions used as colorimetric standards in determining lead; and for the preparation of other lead salts.

Maximum Limits of Impurities

Insoluble Substances	0.0000%
Copper (Cu)	0.0025%
Iron (Fe)	0.0025%
Earths and Alkalis	0.1000%

Methods of Testing

Insoluble Substances (Lead Sulphate).—Dissolve 5 gm. in 50 mls of water. No insoluble residue should remain.

Copper and Iron.—Dissolve 2 gm. in 10 mls of water and add potassium ferrocyanide solution. No reddish or bluish color should be produced.

Earths and Alkalis.—Dissolve 5 gm. in 100 mls of water and pass in hydrogen sulphide gas until the lead is completely precipitated. Filter, evaporate the filtrate to dryness and ignite and weigh the residue. The weight should not exceed 0.005 gm.

**LEAD OXIDE, BROWN, FREE FROM
MANGANESE****(Lead Superoxide; Lead Peroxide; Lead Dioxide)** PbO_2 Mol. Wt. 239.1

Lead oxide, brown, is a dark-brown, amorphous powder, insoluble in water, soluble in hot, dilute hydrochloric acid with evolution of chlorine. It contains at least 90% of PbO_2 .

The method generally preferred for the quantitative determination of lead peroxide is that given under the methods of testing, occasionally with slight modifications introduced by various analysts. An iodometric method said to be excellent is described in Treadwell-Hall's Analytical Chemistry, 2, 675 (1915).

Lead oxide, brown, free from manganese, is used principally in the detection and colorimetric determination of small quantities of manganese, as in iron and steel; for the separation of chlorine and bromine; and in the detection of benzidine, tolidine, aniline, morphine, strychnine and some other alkaloids.

The reagent should be stored in well-stoppered containers.

Maximum Limits of Impurities

Chlorides (Cl)	0.0020%
Sulphates (SO_4)	0.0030%
Manganese (Mn)	0.0002%
Calcium and Alkalis	1.0000%
Water-soluble Substances	0.0500%

Methods of Testing

Chlorides.—Boil 5 gm. of lead peroxide with 60 mls of water and 5 mls of nitric acid, filter and to 30 mls of the filtrate add silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Digest 5 gm. with 30 mls of a cold, saturated solution of sodium bicarbonate for three or four hours, shaking frequently. Filter, acidulate the filtrate with hydrochloric acid, boil the solution ten minutes, add 2 mls

of barium chloride solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Manganese.—Heat 5 gm. with 10 mls of sulphuric acid until completely decomposed. When cold, mix the mass with 20 mls of water and 0.5 gm. of the lead peroxide and warm again. The liquid should not acquire a red color.

Calcium and Alkalis.—Dissolve 1 gm. in 10 mls of hydrochloric acid diluted with 100 mil of water, boil ten minutes to expel the chlorine and supersaturate the liquid with hydrogen sulphide gas. Filter off the precipitate, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.01 gm.

Water-soluble Substances (Lead Nitrate, etc.).—Boil 2 gm. with 50 mls of water, filter, evaporate 25 mls of the filtrate and ignite and weigh the residue. Not more than 0.0005 gm. should be found.

Quantitative Method.—Gently heat about 0.5 gm., accurately weighed, in a porcelain dish with 50 mls of tenth-normal oxalic acid solution and 20 mls of nitric acid until the lead peroxide is completely decomposed and dissolved. Dilute with 200 mls of water, heat the solution to the boiling-point and allow tenth-normal solution of potassium permanganate to run into it until the red color imparted by 1 drop no longer completely disappears within one-half minute. At least 90% of PbO_2 should be found.

1 mil of tenth-normal $\text{H}_2\text{C}_2\text{O}_4 = 0.011955$ gm. of PbO_2 ,
log 07755.

LEAD OXIDE, BROWN, FOR ULTIMATE ANALYSIS

(Lead Superoxide; Lead Peroxide; Lead Dioxide)

PbO_2

Mol. Wt. 239.1

Lead oxide, brown, for ultimate analysis has the physical properties as described under Lead Oxide, Brown, Free from Manganese. It contains at least 90% of PbO_2 .

This reagent is especially intended for use in ultimate organic analysis. Regarding this use see Kingscott and Knight's *Methods of Quantitative Analysis*, 34 (1914).

Maximum Limits of Impurities

Chlorides (Cl)	0.0020%
Sulphates (SO ₃)	0.0010%
Nitrates (N ₂ O ₅)	0.0032%
Carbonates (CO ₂)	0.0000%
Calcium and Alkalis	1.0000%

Methods of Testing

Chlorides, Calcium and Alkalis.—Perform the tests for these impurities as directed under Lead Oxide, Brown, Free from Manganese. The reagent should meet the requirements there stated.

Sulphates.—Digest 25 gm. of the lead peroxide with 50 mls of 20% sodium carbonate solution for one hour on a steam-bath, shaking frequently. Filter, wash the residue with 50 mls of hot water, acidulate the combined filtrate and washings with hydrochloric acid, boil the solution for ten minutes, add 2 mls of barium chloride solution and allow to stand fifteen to twenty-four hours. No precipitate of barium sulphate should form.

Nitrates.—Boil 1 gm. with a mixture of 5 mls of 36% acetic acid and 10 mls of water, filter, add to the filtrate 1 drop of indigo solution and mix the liquid with 10 mls of sulphuric acid. The blue color should persist for a few moments at least after mixing.

Carbonates.—Pour nitric acid over 5 gm. of the lead peroxide. No evolution of gas should be apparent even with the use of a magnifying lens.

Quantitative Method.—Carry out the quantitative determination by the method described under Lead Oxide, Brown, Free from Manganese. At least 90% of PbO₂ should be found.

LEAD OXIDE, RED**(Red Lead; Lead Orthoplumbate)** Pb_3O_4

Mol. Wt. 685.6

Lead oxide, red, occurs as a heavy, orange-red powder, almost insoluble in water but dissolved by an excess of glacial acetic acid. Nitric acid alone does not dissolve it but converts it to lead peroxide, which upon the addition of hydrogen peroxide solution dissolves with the evolution of oxygen. It contains at least 90% of Pb_3O_4 . Red oxide of lead is a combination of the monoxide and the dioxide and frequently contains small amounts of the former uncombined.

The red oxide of lead on exposure to the air absorbs moisture and carbon dioxide. On heating these are expelled, but on continued heating decomposition takes place with the formation of yellow lead oxide and liberation of oxygen. Like lead oxide, yellow, the red oxide is reduced to metallic lead when ignited with organic substances. Platinum should not be used in igniting lead compounds.

Lead oxide, red, is chiefly used in the determination of manganese in iron and steel and in separating iron from nickel, cobalt, copper and lead.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Carbonates	Traces
Lead Silicate and Sulphate	0.5000%
Manganese (Mn)	0.0002%
Water-soluble Impurities	0.5000%

Methods of Testing

Carbonates, Lead Silicate and Sulphate.—Treat 1 gm. with 20 mls of 10% nitric acid. Not more than slight effervescence should result. To the mixture add hydrogen peroxide solution until the brown lead oxide is dissolved, boil the solution for fifteen minutes (replacing the water lost by evaporation), cool and filter. Wash, dry and weigh

the insoluble residue. Its weight should not exceed 0.005 gm.

Manganese.—Heat 5 gm. with 15 mls of sulphuric acid until the oxide is completely decomposed. Cool, mix the mass with 15 mls of water, add 0.5 gm. of the lead oxide and warm the mixture. The liquid portion should not acquire a red color.

Water-soluble Impurities.—Mix 1 gm. with 50 mls of water, heat the mixture to boiling, cool, filter, evaporate the filtrate to dryness and weigh the residue. The weight should not exceed 0.005 gm.

Quantitative Method.—Accurately weigh about 1 gm. of the lead oxide, add it to a cool solution of 40 gm. of sodium acetate and 2 gm. of potassium iodide in 50 mls of 50% acetic acid, agitate the mixture until the oxide has dissolved and then titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 90% of Pb_2O_4 should be found.

1 mil of tenth-normal $Na_2S_2O_3 = 0.03428$ gm. of Pb_2O_4 ,
log 53504.

LEAD OXIDE, YELLOW

(Litharge)

PbO

Mol. Wt. 223.2

Lead oxide, yellow, occurs as a yellow or reddish-yellow powder, almost insoluble in water to which, however, it imparts a slightly alkaline reaction. It is soluble in nitric or acetic acid and in warm solutions of sodium or potassium hydroxide.

On exposure to the air the lead oxide gradually absorbs moisture and carbon dioxide, which are expelled upon ignition. Ignited in the presence of organic matter the lead is reduced to the metallic state. Platinum dishes should not be used when heating lead or any of its compounds, as an alloy destructive to the platinum is easily formed.

The reagent is used chiefly in the analysis of organic substances containing halogens; as a flux in the fusion of minerals; in the saponification of fats; and for preparing lead subacetate solution.

Lead oxide should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Substances Insoluble in Acetic Acid	0.2500%
Volatile Substances	0.2500%
Copper (Cu)	0.0010%
Iron (Fe)	0.0020%
Aluminum (Al)	0.1000%
Nitrates and Nitrites (as N_2O_5)	0.0160%
Chlorides (Cl)	0.0050%
Earths and Alkalis	0.3000%

Methods of Testing

Substances Insoluble in Acetic Acid.—Mix 2 gm. of the lead oxide with 5 mls of water, add 10 mls of 36% acetic acid, boil the mixture for several minutes, cool and filter. Wash the undissolved residue, dry it at 100° C. and weigh it. The weight should not exceed 0.005 gm.

Volatile Substances (Carbon Dioxide, Water).—Heat 2 gm. to incipient fusion and determine the loss in weight. The loss should not exceed 0.005 gm.

Copper, Iron and Aluminum.—Mix 2 gm. with 15 mls of water and add 10 mls of nitric acid to effect solution. Then add 3 mls of sulphuric acid, filter off the resulting precipitate and add to the filtrate a slight excess of ammonia water. The liquid should not acquire a blue color nor should a precipitate form.

Nitrates and Nitrites.—Mix 1 gm. with 5 mls of water and add 5 mls of 36% acetic acid to effect solution. Add 0.2 ml of indigo solution and 10 mls of sulphuric acid and agitate the mixture. A blue color should persist after the mixing.

Chlorides.—Dissolve 1 gm. in 20 mls of water with the aid of 5 mls of nitric acid and add silver nitrate solution. Not more than a slight opalescence should result.

Earths and Alkalis.—Dissolve 1 gm. in 10 mls of 36% acetic acid, dilute the solution with 50 mls of water and precipitate the lead completely with hydrogen sulphide gas. Filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.003 gm.

LEAD SUBACETATE SOLUTION

Lead subacetate solution is a clear, colorless liquid, having a specific gravity of about 1.25 and containing 18% to 20% of Pb in the form of basic acetate. It is alkaline to litmus paper but does not redden phenolphthalein.

Lead subacetate solution is used principally as a precipitant and clarifier in urine analysis and organic analysis and in the detection of benzoic and formic acids, glucose, cottonseed oil and carbon monoxide blood; also for differentiating dioxybenzenes.

When exposed to the air the solution takes up carbon dioxide and soon becomes cloudy from the formation of lead carbonate. It should be stored in small, well-stoppered containers.

Maximum Limits of Impurities

Copper (Cu)	0.0006%
Iron (Fe)	0.0002%

Methods of Testing

Copper and Iron.—Acidify 10 mls of the solution with 2 mls of 36% acetic acid and add potassium ferrocyanide solution. The precipitate which forms should be white, untinged with red or blue.

Quantitative Method.—Place in a 200-ml flask about 1.5 gm. of the solution, accurately weighed, dilute it with 50 mls of water, free from carbon dioxide, add 50 mls of tenth-normal oxalic acid solution, and, after thoroughly mixing, fill the flask to the mark with water. Filter, rejecting the first 20 mls of filtrate, and to the next 100 mls add 5 mls of sulphuric acid, warm to about 70° C. and titrate with tenth-normal potassium permanganate solution.

1 ml of tenth-normal $\text{H}_2\text{C}_2\text{O}_4 = 0.01036$ gm. of Pb,
log 01536.

LIME, CHLORINATED**(Calcium Hypochlorite)**

Chlorinated lime is usually obtained as a white or grayish-white, granular powder, having a chlorine-like odor. It is sometimes prepared in the form of cubical pieces adapted to use in gas generating apparatus for obtaining chlorine in the laboratory. The reagent is partially soluble in water, leaving a residue of calcium hydroxide. It contains at least 25% of available Cl.

Chlorinated lime is very susceptible to change on exposure to air and moisture, soon losing its active chlorine content. Acids at once liberate chlorine, and even carbon dioxide gradually effects the same decomposition. Contact with organic matter causes deterioration.

Chlorinated lime is chiefly used as a source of chlorine and as an oxidizing and bleaching agent; for the detection of aniline, acetanilid, xanthine, quinine, morphine and emetine; in urinalysis for detecting albumin, indican and bile pigments and in estimating urea; and for the preparation of solutions of alkaline hypochlorites, which are sometimes preferred to the solution of chlorinated lime as a reagent on account of greater stability.

The reagent should be kept in air-tight containers in a cool, dry place.

Method of Testing

Quantitative Method.—Weigh a bottle containing 10 mls of water, then introduce from 3 to 4 gm. of the chlorinated lime and weigh again. Triturate the contents of the weighing bottle thoroughly with 50 mls of water, rinse it into a graduated liter flask, fill with water to the mark and allow it to stand ten minutes. Mix thoroughly, measure off 100 mls of the mixture, add to it 1 gm. of potassium iodide and 5 mls of 36% acetic acid, and titrate with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 25% of available Cl should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.003546$ gm. of Cl,
log 54974.

LIME WATER**(Calcium Hydroxide Solution)**

Lime water is a clear, colorless liquid of alkaline reaction, containing in 100 mls at least 0.15 gm. of $\text{Ca}(\text{OH})_2$. It consists of a solution of calcium hydroxide made by slaking lime and, after washing it by decantation to remove alkalis and barium and strontium hydroxides, making a saturated solution of the residue. On exposure to the air the reagent soon becomes turbid, due to absorption of carbon dioxide.

Lime water is used as an alkalizing agent; in detecting citric and tartaric acids and carbon dioxide; and in testing eugenol.

The reagent should be stored in small, well-stoppered containers.

Method of Testing

Quantitative Method.—Titrate the lime water with standard acid solution, using methyl orange as indicator. At least 0.15 gm. of $\text{Ca}(\text{OH})_2$ per 100 mls should be found.

1 mil of normal HCl = 0.037053 gm. of $\text{Ca}(\text{OH})_2$,
log 56882.

MAGNESIA MIXTURE

Magnesia mixture is a clear, colorless liquid having the odor of ammonia. It may be prepared by dissolving 5.5 gm. of magnesium chloride and 7 gm. of ammonium chloride in 65 mls of water and adding to the solution 35 mls of 10% ammonia water. The mixture should be set aside for a few days and then filtered and should be perfectly clear when used.

Magnesia mixture serves to precipitate orthophosphates and arsenates as ammonium-magnesium double salts for both qualitative and quantitative purposes. The reagent should be stored in well-stoppered bottles.

MAGNESIUM CARBONATE**(Basic Magnesium Carbonate)**

Magnesium carbonate occurs usually as a white, bulky powder or light friable masses, soluble in acids with effervescence. It has no definite composition, but contains varying amounts of magnesium carbonate, magnesium hydroxide and water, and yields on ignition at least 35% of residue (MgO).

Magnesium carbonate is practically insoluble in carbon dioxide-free water although it imparts to it an alkaline reaction; in carbonated water and in solutions of ammonium salts it dissolves easily. From solutions in the latter it is not precipitated by boiling but separates on the addition of caustic alkalis.

The principal use of the reagent is in the preparation of magnesium salts.

Maximum Limits of Impurities

Water-soluble Substances	0.7500%
Substances Insoluble in Hydrochloric Acid	0.0000%
Sulphates (SO ₄)	0.0125%
Barium (Ba)	0.0150%
Calcium (Ca)	0.0600%
Aluminum (Al)	0.0500%
Iron (Fe)	0.0150%
Other Heavy Metals	0.0000%

Methods of Testing

Water-soluble Substances.—Powder the magnesium carbonate if it is not already a powder, suspend 3 gm. in 90 mls of water, heat to boiling and filter while hot. Evaporate 60 mls of the filtrate and weigh the residue. The weight should not exceed 0.015 gm.

Substances Insoluble in Hydrochloric Acid.—Dissolve 1 gm. in a mixture of 4 mls of hydrochloric acid and 16 mls of water. The solution should be clear and colorless.

Sulphates.—To the solution obtained in the preceding test add barium chloride solution. No change should be apparent.

Barium.—Dissolve 1 gm. in a mixture of 4 mls of hydrochloric acid and 16 mls of water and add diluted sulphuric acid. No turbidity should be produced.

Calcium.—Dissolve 1 gm. in a mixture of 10 mls of 36% acetic acid and 30 mls of water, boil the solution, add 20 mls of ammonia water and some ammonium oxalate solution and allow to stand five minutes. No turbidity should result.

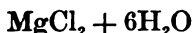
Aluminum.—Dissolve 1 gm. in a mixture of 10 mls of acetic acid and 40 mls of water, boil off the carbon dioxide, add 10 mls of ammonia water and heat on the steam-bath until the odor of ammonia has almost or entirely disappeared. Then let it stand until cool. No flocculent precipitate should form.

Iron.—Dissolve 0.5 gm. in a mixture of 2 mls of hydrochloric acid and 20 mls of water, add 0.5 ml of potassium ferrocyanide solution and allow to stand one minute. No blue color should develop.

Other Heavy Metals.—Dissolve 1 gm. in a mixture of 10 mls of 36% acetic acid and 40 mls of water and add hydrogen sulphide water. No change should be apparent.

Determination of Residue on Ignition.—Ignite an accurately weighed portion of the magnesium carbonate to constant weight. The weight of the residue should correspond to at least 35% of the sample taken.

MAGNESIUM CHLORIDE



Mol. Wt. 203.34

Magnesium chloride is usually in the form of white, deliquescent crystals, soluble in about 0.6 part of water and in about 2 parts of alcohol. The aqueous solution is neutral

to litmus. On being heated a little above 100° C. the salt melts, loses hydrochloric acid and water and is finally changed to the oxide.

Magnesium chloride is used chiefly in the preparation of magnesia mixture. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Substances Insoluble in Alcohol	0.0000%
Sulphates (SO_4)	0.0100%
Phosphates (P_2O_5)	0.0020%
Arsenates (As_2O_5)	0.0100%
Ammonium Salts (NH_4)	0.0300%
Barium (Ba)	0.0020%
Heavy Metals	0.0000%
Calcium (Ca)	0.0050%

Methods of Testing

Substances Insoluble in Alcohol.—Dissolve 1 gm. in 10 mls of alcohol of 75% strength (by volume). The salt should dissolve clearly without leaving a residue.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add 1 mil of hydrochloric acid and some barium chloride solution. No change should be apparent.

Phosphates and Arsenates.—Dissolve 2.5 gm. of magnesium chloride and 5 gm. of ammonium chloride in 20 mls of water, add 10 mls of ammonia water and allow it to stand twelve to eighteen hours. No precipitate should form.

Ammonium Salts.—Heat 3 gm. with 10 mls of water and 2 mls of 27% sodium hydroxide solution. No odor of ammonia should be perceptible in the vapors evolved.

Barium.—Dissolve 1 gm. in 20 mls of water and add a few drops of diluted sulphuric acid. No change should be apparent.

Calcium and Heavy Metals.—Dissolve 1 gm. of magnesium chloride and 1 gm. of ammonium chloride in 20 mls of water. Add 5 mls of ammonia water and then, in suc-

cession, ammonium oxalate solution and a few drops of ammonium sulphide solution. No change should be apparent after the addition of either reagent.

Dissolve 1 gm. in 20 mls of water, add a few drops of hydrochloric acid and some hydrogen sulphide water. No change should be apparent.

MAGNESIUM OXIDE

(Magnesia)

MgO

Mol. Wt. 40.32

Magnesium oxide is a white, light, fine powder, almost insoluble in water, insoluble in alcohol, but readily soluble in dilute acids. It contains at least 80% of MgO. On exposure to the air it gradually absorbs moisture and carbon dioxide, both of which may be expelled by ignition. Small amounts of carbonates and moisture are frequently present in the reagent.

The oxide is used principally as a neutralizing agent and for the preparation of magnesium salts. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Water-soluble Substances	0.7500%
Substances Insoluble in Hydrochloric Acid	0.0000%
Sulphates (SO ₄)	0.0250%
Chlorides (Cl)	0.0100%
Carbonates	Traces
Nitrates (N ₂ O ₅)	0.0064%
Barium (Ba)	0.0200%
Calcium (Ca)	0.0200%
Iron (Fe)	0.0250%
Other Heavy Metals	0.0000%

Water-soluble Substances.—Heat to boiling a mixture of 3 gm. of magnesium oxide and 75 mls of water, filter while hot, evaporate 50 mls of the filtrate and weigh the residue. The weight should not exceed 0.015 gm.

Substances Insoluble in Hydrochloric Acid.—Dissolve 1 gm. in 20 mls of 10% hydrochloric acid. The solution should be clear.

Sulphates.—To the solution obtained in the preceding test add barium chloride solution. Not more than a slight opalescence should be produced.

Chlorides.—Dissolve 0.5 gm. in 20 mls of 10% nitric acid and add silver nitrate solution. Not more than a slight opalescence should be produced.

Carbonates.—Heat to boiling a mixture of 0.1 gm. of magnesium oxide with 5 mls of water, cool the mixture and pour it into 5 mls of 36% acetic acid. Solution should take place without the evolution of more than a few isolated gas bubbles.

Nitrates.—Dissolve 0.5 gm. in 15 mls of 10% sulphuric acid, add a few milligrams of sodium chloride and 1 drop of indigo solution and mix with 10 mls of concentrated sulphuric acid. A blue color should persist after mixing.

Barium.—Dissolve 1 gm. in 20 mls of 10% hydrochloric acid and add 1 or 2 mls of 10% sulphuric acid. No change should be apparent.

Calcium.—Thoroughly shake 1 gm. of magnesium oxide with 20 mls of water for three or four minutes, filter and to the filtrate add ammonium oxalate solution. No turbidity should result.

Iron.—Dissolve 0.2 gm. in 10 mls of 5% hydrochloric acid and add 0.5 ml of potassium ferrocyanide solution. No blue color should be produced.

Other Heavy Metals.—Dissolve 0.5 gm. in 20 mls of water, acidulated with 2 to 3 mls of hydrochloric acid and add hydrogen sulphide water. No change should be apparent.

Quantitative Method.—Dissolve a weighed sample of the oxide in an excess of standard acid solution and titrate the excess acid with standard alkali solution, using methyl orange as indicator. At least 80% of MgO should be found.

1 ml of normal HCl = 0.02016 gm. of MgO, log 30449.

MAGNESIUM OXIDE, FREE FROM SULPHATES**MgO**

Mol. Wt. 40.32

Magnesium oxide, free from sulphates, has the appearance, solubilities and properties noted under Magnesium Oxide. It contains at least 80% of MgO.

The reagent is suitable for the usual purposes for which Magnesium Oxide is used and is especially adapted to the determination of sulphur in iron, steel, organic and other substances. For sulphur tests it is often combined with sodium carbonate as in Eschka's mixture, described on page 208.

The oxide should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Substances Soluble in Water	0.7500%
Substances Insoluble in Hydrochloric Acid	0.0000%
Sulphates (SO ₄)	0.0050%
Chlorides (Cl)	0.0100%
Carbonates	Traces
Nitrates (N ₂ O ₅)	0.0064%
Barium (Ba)	0.0200%
Calcium (Ca)	0.0200%
Iron (Fe)	0.0250%
Other Heavy Metals	0.0000%

Methods of Testing

Sulphates.—Dissolve 5 gm. in 25 mls of water with the aid of 25 mls of hydrochloric acid, dilute the solution to 100 mls, boil it, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Other Impurities.—Perform the tests for the other impurities listed above as directed under Magnesium Oxide. The results should be as there stated.

Quantitative Method.—Dissolve and titrate the oxide as directed under Magnesium Oxide. At least 80% of MgO should be found.

1 mil of normal HCl = 0.02016 gm. of MgO, log 30449.

MAGNESIUM OXIDE AND SODIUM CARBONATE

(Eschka's Mixture)

Eschka's mixture is a white powder or small, soft granules, consisting of a mixture of 2 parts of magnesium oxide with 1 part of dried sodium carbonate. It is partly soluble in water; completely soluble in acids with effervescence.

Eschka's mixture is employed chiefly in the determination of sulphur in coal, coke and other organic bodies. It should be kept in well-stoppered containers.

Maximum Limit of Impurity

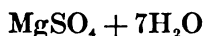
Sulphate (SO_4) 0.0025%

Method of Testing

Sulphates.—Dissolve 10 gm. of Eschka's mixture in 60 mls of water with the aid of 40 mls of hydrochloric acid, heat to boiling, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

MAGNESIUM SULPHATE

(Epsom Salt)



Mol. Wt. 246.5

Magnesium sulphate is usually in the form of small, clear, colorless, prismatic crystals. It is soluble in about 1 part of water and its solutions are neutral to litmus. It is somewhat efflorescent.

The salt is used principally for the preparation of magnesia mixture and in separating globulin from albumin. It should be kept in tightly stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0010%
Phosphates (P_2O_5)	0.0016%
Iron (Fe)	0.0010%
Other Heavy Metals	0.0000%
Arsenic (As)	0.0015%

Methods of Testing

Chlorides.—Dissolve 1 gm. of magnesium sulphate in 20 mils of water and add 1 mil of nitric acid and some silver nitrate solution. No change should be apparent.

Phosphates.—Dissolve 3 gm. of magnesium sulphate and 6 gm. of ammonium chloride in 24 mils of water, add 12 mils of ammonia water and allow it to stand twelve to eighteen hours. No precipitate should form.

Iron.—Dissolve 1 gm. in 20 mils of water, add a few drops of nitric acid, boil the solution and add potassium sulphocyanate solution. No red color should be produced.

Other Heavy Metals.—Dissolve 1 gm. each of magnesium sulphate and ammonium chloride in 20 mils of water, acidulate with 1 mil of hydrochloric acid and add hydrogen sulphide water. No change should be apparent. Then make the solution alkaline with ammonia water. No color or precipitate should be produced.

Arsenic.—Mix 1 gm. of powdered magnesium sulphate with 3 mils of stannous chloride solution and allow it to stand for one hour. No dark color should develop.

MAGNESIUM AND AMMONIUM CHLORIDE



Mol. Wt. 256.84

Magnesium and ammonium chloride is usually in the form of white crystals or a crystalline powder, soluble in about 6 parts of water. It is deliquescent in moist air. The salt is used principally for the preparation of magnesia mixture employed for the determination of phosphates and arsenates. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0100%
Heavy Metals	0.0000%
Calcium (Ca)	0.0050%
Barium (Ba)	0.0020%
Phosphates (P_2O_5)	0.0010%
Arsenates (As_2O_5)	0.0050%

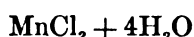
Methods of Testing

Sulphates, Heavy Metals, Calcium and Barium.—Apply the tests for these impurities as directed under Magnesium Chloride. The results should be as there stated.

Phosphates and Arsenates.—Dissolve 5 gm. in 10 mls of water, add 5 mls of ammonia water and allow to stand twelve to eighteen hours. No precipitate should form.

MANGANESE CHLORIDE

(Manganous Chloride)



Mol. Wt. 197.91

Manganese chloride is usually in the form of reddish, hygroscopic crystals, soluble in about 1 part of water; also soluble in alcohol. On warming to about 50° C. the salt melts. The reagent is used chiefly in the determination of oxygen and boric acid. It should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Sulphates (SO ₃)	0.0100%
Calcium (Ca)	0.0200%
Salts of Magnesium and the Alkalis	0.1000%
Iron (Fe)	0.0008%
Zinc (Zn)	0.0500%
Other Heavy Metals	0.0000%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 20 mls of water and add 1 mil of hydrochloric acid and some barium chloride solution. No turbidity should result.

Calcium.—Dissolve 1 gm. in 20 mls of water and add 2 mls of ammonium oxalate solution. No turbidity should be produced within one minute. (A crystalline precipitate of manganous oxalate may form on standing for some time.)

Salts of Magnesium and the Alkalis.—Dissolve 2 gm. in 40 mls of water, precipitate the manganese by adding sufficient 10% solution of ammonium carbonate, filter, evaporate the filtrate and ignite and weigh the residue. Not more than 0.002 gm. should be found.

Iron.—Dissolve 1 gm. in 20 mls of water, add 1 mil of hydrochloric acid and 3 mls of chlorine water and heat the mixture to boiling. Then cool the solution and add potassium sulphocyanate solution. No red color should result.

Zinc.—Dissolve 1 gm. each of manganese chloride and sodium acetate in 10 mls of water and add a few drops of 36% acetic acid and of hydrogen sulphide water. No turbidity due to zinc sulphide should result.

Other Foreign Metals.—Dissolve 1 gm. in 20 mls of water and add 2 to 3 mls of hydrochloric acid and some hydrogen sulphide water. No change should be apparent.

MANGANESE DIOXIDE

(Manganese Peroxide; Manganese Superoxide)

MnO₂

Mol. Wt. 86.93

Manganese dioxide usually occurs as a fine, heavy, black powder. It is insoluble in water but is soluble in hydrochloric acid, chlorine being evolved on warming. It contains at least 85% of MnO₂. Other oxides of manganese are generally present, some of which may be reduced and estimated as MnO₂ by the quantitative method given below. This is of no significance as the value of the reagent depends only upon its available oxygen.

Manganese dioxide is used chiefly as an oxidizing agent; for preparing chlorine and oxygen; in detecting strychnine, brucine, apocodeine, aniline and artificial coloring in red wines.

Method of Testing

Quantitative Method.—Dissolve about 0.2 gm., accurately weighed, in a mixture of 50 mls of tenth-normal

oxalic acid solution and 3 mls of sulphuric acid, heat to about 80° C. and titrate the excess of oxalic acid with tenth-normal solution of potassium permanganate. At least 85% of MnO_2 should be found.

1 mil of tenth-normal $\text{H}_2\text{C}_2\text{O}_4 = 0.0043465$ gm. of MnO_2 ,
log 63814.

MANGANESE SULPHATE

(Manganous Sulphate)

$\text{MnSO}_4 + 4\text{H}_2\text{O}$

Mol. Wt. 223.05

Manganese sulphate occurs usually as pale red crystals, soluble in less than 1 part of water but insoluble in alcohol. The aqueous solution is neutral to litmus. The salt is somewhat efflorescent when exposed to the air. On ignition it loses its water and is converted to anhydrous manganous sulphate; in this way the percentage of water in the crystals (theory 32.29%) may be easily determined, thus differentiating this salt from the several other manganous sulphates possessing varying amounts of water of crystallization.

Manganese sulphate is chiefly used in permanganate titrations in the presence of hydrochloric acid; in the determination of oxygen and of boric acid; and in the detection of cinnamic acid.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Substances Reducing Permanganate	Trace
Calcium (Ca)	0.0200%
Iron (Fe)	0.0008%
Zinc (Zn)	0.0500%
Other Foreign Metals	0.0000%
Salts of Magnesium and the Alkalis	0.1000%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 1 mil of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Substances Reducing Permanganate.—Dissolve 10 gm. in 200 mls of water, add 3 mls each of sulphuric acid and phosphoric acid and just sufficient tenth-normal solution of potassium permanganate to impart a slight red color to the solution. Not more than 0.1 ml should be required.

Calcium, Iron, Zinc, Other Foreign Metals and Salts of Magnesium and the Alkalis.—Perform the tests for these substances as directed under Manganese Chloride. The results should be as there indicated.

MANNITE

(Mannitol; Dextromannite)

$C_6H_{14}O_6$

Mol. Wt. 182.14

Mannite usually occurs as fine, white, needle crystals, sometimes molded into blocks or irregular pieces. It is soluble in about 6 parts of water, and in about 1,000 parts of alcohol; readily soluble in boiling alcohol but insoluble in ether. It melts at about 166° C. (theory 169° C. corr.) and sublimates when slowly heated. Its solutions are neutral to litmus.

Mannite is a hexahydric alcohol and somewhat resembles the sugars in some of its properties. It has a sweet taste and is strongly dextrorotary in the presence of borax, although practically inactive in solution alone. It does not reduce alkaline copper solutions.

The reagent is chiefly used in titrating boric acid and as a constituent of copper solutions employed in the detection and determination of reducing sugars.

Maximum Limits of Impurities

Ash	0.1000%
Reducing Substances	0.0000%

Methods of Testing

Ash.—Ignite 1 gm. of mannite and weigh the ash. The weight should not exceed 0.001 gm.

Reducing Substances.—Dissolve 5 gm. in 25 mls of warm water, add 2 to 3 mls of hydrochloric acid and heat the solution to boiling. Cool, make slightly alkaline with sodium hydroxide, add 10 mls of Fehling's solution, boil the mixture two to three minutes and allow it to stand half an hour. No deposit of cuprous oxide should form.

MERCURY

Hg

Mol. Wt. 200.6

Mercury for reagent use is a silver-white liquid, boiling at about 357° C. (theory 357.33° C.). When pure it congeals at —38.85° C. and has a specific gravity of 13.5953 at 0°/4° C. Mercury is very slowly but perceptibly volatile even at ordinary temperatures, a fact to be considered in the accurate weighing of small amounts, such as those obtained in analytic operations.

It is used in the determination of nitrogen by the Kjeldahl method; for the preparation of mercury salts and solutions, such as Millon's solution of mercury nitrate; as a catalyzer; and as a cathode in electro-analysis for which purpose it is well suited on account of the ease with which it amalgamates other metals.

Mercury should be stored in strong, tightly-stoppered containers.

Maximum Limits of Impurities

Foreign Metals	Traces
Insoluble Matter	0.0000%
Non-volatile Matter	0.0025%

Methods of Testing

Foreign Metals.—(a) The bright surface of the mercury should not be affected by shaking with pure air.

(b) Overlay a solution of 4.5 gm. of sodium thiosulphate in 5 mls of water upon 5 gm. of the mercury and apply heat so that the aqueous solution boils for about one minute. (An open flame is apt to overheat. Immerse the container in an oil- or salt-bath.) The mercury should not lose its

luster and should not acquire more than a slight yellowish color.

Insoluble Matter.—Mercury should dissolve completely in nitric acid.

Non-volatile Matter.—Ignite 20 gm. of mercury in a porcelain dish. Not more than 0.0005 gm. of residue remains.

MERCURY BICHLORIDE

(Mercuric Chloride)

HgCl₂

Mol. Wt. 271.52

Mercury bichloride is usually in the form of white, translucent crystals. It is soluble in about 16 parts of water and in about 3 parts of alcohol; also soluble in ether. The salt contains at least 99.5% of HgCl₂.

The solubility of the salt in ether depends upon the amount of alcohol or water contained in the ether. Although mercury bichloride is scarcely soluble in absolute ether, it is more soluble in ether saturated with water than in water alone. Hydrochloric acid or chlorides of the alkalis increase the solubility of the salt in water. The aqueous solution has an acid reaction to litmus but the addition of sufficient sodium chloride renders it neutral to that indicator.

Mercury bichloride is volatilized slowly from its aqueous solution on heating. Aqueous solutions slowly decompose in the dark with separation of mercuric oxide and in the light with formation of mercurous chloride. This decomposition is retarded by the presence of hydrochloric acid or alkali chlorides, and also by tartaric or citric acid.

Mercury bichloride is chiefly used for the detection and often in the determination of substances which exert a reducing action upon it, such as stannous salts, metallic iron, formic acid, formaldehyde, etc.; for the detection and determination of small quantities of arsenic; for detecting sodium carbonate in the bicarbonate; as a reagent for alkaloids, albuminoids, and gelatin; for detecting sulphur and

phosphorus compounds in acetylene; for determining the iodine number of fats; and in the preparation of various mercurial reagents.

The reagent should be stored and handled with due regard for its poisonous nature.

Maximum Limits of Impurities

Foreign Matter not Precipitated by Hydrogen Sulphide	0.0100%
Arsenic (As)	0.0080%
Mercurous Chloride and Other Substances Insoluble in Ether	0.0000%

Methods of Testing

Foreign Matter not Precipitated by Hydrogen Sulphide.—Dissolve 5 gm. of mercury bichloride in 100 mls of water, add a few drops of hydrochloric acid and pass hydrogen sulphide gas into the solution until the mercury is completely precipitated. Filter, evaporate the filtrate to dryness, gently ignite the residue and weigh it. Not more than 0.0005 gm. should be found.

Arsenic.—Shake the mercury sulphide obtained in the above test with a mixture of 5 mls of ammonia water and 45 mls of water, filter, and acidulate the filtrate with hydrochloric acid. A yellow color or precipitate should not be produced.

Mercurous Chloride and Other Substances Insoluble in Ether.—Dissolve 1 gm. in 25 mls of ether. No insoluble residue should remain.

Quantitative Method.—Dry the salt to constant weight in a desiccator over sulphuric acid, dissolve 0.3 to 0.4 gm., accurately weighed, in 10 mls of water and transfer the solution to the cathode cup of an electrolytic apparatus, previously weighed with its metallic mercury. Dilute the solution to about 20 mls, add 1 ml of nitric acid and 10 mls of toluol and pass through the solution a current of 1 to 3 amperes and 8 to 12 volts, stirring the solution by rotating the anode about 400 revolutions per minute. After

about twenty minutes, when the mercury is all removed from solution (to be ascertained by testing a few drops of the solution with hydrogen sulphide) wash the mercury with water by siphonation without interrupting the current, and when the current has dropped to nearly zero, remove the cup and wash the mercury with alcohol and then with ether, dry it in a desiccator over sulphuric acid and weigh. An increase in weight equivalent to at least 99.5% of HgCl_2 should be found.

$$\text{Hg} \times 1.35354 = \text{HgCl}_2, \log 13147.$$

Mercury bichloride may also be determined gravimetrically by precipitation as the sulphide. Volumetric methods are described in Chem. Abst., 4, 1084 (1910), and 8, 1721 (1914).

MERCURY BROMIDE

(Mercuric Bromide)

HgBr_2

Mol. Wt. 360.44

Mercury bromide usually occurs as a white crystalline powder, soluble in about 200 parts of cold water and in about 25 parts of boiling water; readily soluble in boiling alcohol. It is more readily soluble in solutions of alkali bromides than in water alone. Pure mercuric bromide is fairly stable but when it contains traces of mercurous salt, as it nearly always does, or of organic matter, it darkens slightly on exposure to light.

The reagent is used principally in detecting and determining small quantities of arsenic. For this purpose the U. S. Pharmacopœia, 9th Revision, employs an alcoholic solution containing 5 gm. of mercury bromide in 100 mls. Strips of paper moistened with this solution and then dried are used in the test which is minutely described on page 584 of the Pharmacopœia. See also Scott's Standard Methods of Chemical Analysis, 2d ed., revised, 40 (1918), wherein standard arsenic stains are pictured and the application of the Gutzeit test to various materials is described.

Mercury bromide should be stored in well-stoppered bottles of amber-colored glass.

Maximum Limits of Impurities

Insoluble Matter	Trace
Non-volatile Matter	0.0500%
Arsenic (As)	0.0002%

Methods of Testing

Insoluble Matter.—Dissolve 2 gm. in 100 mls of 2% solution of potassium bromide. At most a trace of insoluble matter should remain.

Non-volatile Matter.—Ignite 2 gm. of mercury bromide and weigh the residue. The weight should not exceed 0.001 gm.

Arsenic.—Dissolve 5 gm. with the aid of heat in 100 mls of water containing 5 mls of hydrochloric acid and precipitate the mercury completely by passing a current of hydrogen sulphide gas. Filter, wash the precipitate with a little water and then digest it half an hour, with frequent shaking, in a mixture of 5 mls of ammonia water and 45 mls of water. Again filter, acidify the filtrate with sulphuric acid, introduce it into a Marsh apparatus and allow the action to proceed one hour. No deposit of arsenic should be visible in the reduction tube.

MERCURY IODIDE, RED**(Mercuric Iodide)** HgI_2

Mol. Wt. 454.44

Mercury iodide, red, is a scarlet red, amorphous powder, almost insoluble in water but rendered soluble by the presence of soluble iodides. It dissolves in about 100 parts of alcohol or ether.

When heated to about 150° C. the salt becomes yellow and on cooling again assumes its original color. At a higher heat it melts and finally volatilizes. Light does not readily affect it, but if traces of mercurous salts or of organic matter are present some reduction takes place when exposed to light. In alkaline solution it is readily reduced by sugars, aldehydes and other organic substances.

The reagent is used principally for the preparation of Mayer's reagent for alkaloids and Nessler's for ammonia; in the detection of protein, glycogen, glucose and methyl alcohol; and in separating minerals.

The salt should be stored in well-stoppered bottles of amber-colored glass.

Maximum Limits of Impurities

Residue on Ignition	0.2000%
Insoluble Substances	0.2000%
Soluble Mercury Salts	Trace

Methods of Testing

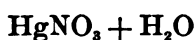
Residue on Ignition.—Ignite 1 gm. at a low red heat and weigh the residue. The weight should not exceed 0.002 gm.

Insoluble Matter.—Dissolve 2 gm. in 100 mls of 2% potassium iodide solution and collect, dry and weigh any insoluble matter. The weight should not exceed 0.004 gm.

Soluble Mercury Salts.—Agitate thoroughly 0.5 gm. with 10 mls of water, filter and test the filtrate with hydrogen sulphide water. Not more than a slight color should result.

MERCURY NITRATE

(Mercurous Nitrate)



Mol. Wt. 280.63

Mercury nitrate usually occurs as colorless crystals. It is soluble in about 2 parts of warm water but the addition of more water precipitates a basic salt. Water acidulated with a little nitric acid dissolves the salt best. It contains at least 96.5% of $\text{HgNO}_3 + \text{H}_2\text{O}$.

Solutions of mercury nitrate are fairly stable if kept in the dark, but exposure to daylight causes the formation of nitrate and a mercuric salt. Heating its solution effects oxidation to mercuric nitrate with liberation of mercury; this change is appreciable at temperatures even as low as 40° C.

Mercury nitrate is used principally in the detection of saccharin, phenol, leucin, brucine, ammonia and some easily oxidizable substances, for example, formic acid.

The reagent should be stored in glass-stoppered bottles of amber glass.

Maximum Limits of Impurities

Non-volatile Matter	0.0250%
Mercuric Salts	Traces

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the salt and weigh the residue. The weight should not exceed 0.0005 gm.

Mercuric Salts.—Dissolve 1 gm. in 5 mls of water acidulated with 3 to 5 drops of nitric acid, dilute the solution with 15 mls of water and add 1 mil of hydrochloric acid. Filter and to the filtrate add hydrogen sulphide water. Not more than traces of precipitate should be produced.

Quantitative Method.—Dissolve about 0.4 gm. of the salt, accurately weighed, in 5 mls of water acidulated with 1 mil of nitric acid, transfer the solution to the cathode cup of an electrolytic apparatus, previously weighed with its metallic mercury, dilute to about 30 mls and electrolyze with a current of 1.5 to 2 amperes and 7 to 10 volts, rotating the anode at the rate of about 500 revolutions per minute. After twenty minutes, when the mercury is all removed from the solution (to be ascertained by testing a few drops with hydrogen sulphide), wash the mercury with water by siphonation without interrupting the current until the current drops to nearly zero. Remove the cathode cup and wash the mercury with water, alcohol and ether in succession, dry it in a desiccator over sulphuric acid and weigh. The gain in the weight should be at least 69% of the sample of mercury nitrate taken, equivalent to at least 96.5% of $\text{HgNO}_3 + \text{H}_2\text{O}$.

MERCURY OXIDE, RED**(Red Mercuric Oxide)****HgO****Mol. Wt. 216.6**

Red mercuric oxide is an orange-red, crystalline powder. It is almost insoluble in water, but is easily soluble in dilute nitric or hydrochloric acid. The solution in nitric acid is clear while that in hydrochloric acid is often slightly turbid. The reagent in dry condition contains at least 99.5% of HgO.

Exposure to sunlight causes darkening of the mercuric oxide, due to reduction to the black mercurous oxide, and sometimes to metallic mercury, oxygen being liberated simultaneously. This decomposition by light accounts for the slight turbidity frequently found in hydrochloric acid solutions of the oxide, the mercurous oxide present forming with the acid the insoluble mercurous chloride.

Red mercuric oxide is used mainly in ultimate analysis as a source of oxygen; in reagents for detecting and determining urea and dextrose; in Deniges' reagent, used in detecting acetone, cystin, citric acid, thiophene, diethylbarbituric acid, etc.; and in the Kjeldahl nitrogen determination.

The reagent should be kept in well-stoppered amber-colored bottles and should be exposed to light as little as possible.

Maximum Limits of Impurities

Non-volatile Matter	0.0250%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0350%
Nitrates (N ₂ O ₅)	0.0160%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the mercuric oxide. Not more than 0.0005 gm. of residue should remain.

Chlorides.—Dissolve 1 gm. in a mixture of 5 mils of nitric acid and 15 mils of water and add silver nitrate solution. Not more than a slight opalescence should result.

Sulphates.—Dissolve 1 gm. in a mixture of 5 mls of nitric acid and 20 mls of water, add barium nitrate solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Nitrates.—Dissolve 1 gm. in a mixture of 2 mls of water and 2 mls of sulphuric acid, and when cold, overlay the solution with ferrous sulphate solution. No color should develop at the plane of contact of the liquids.

Quantitative Method.—Dry the mercuric oxide at 150°, dissolve about 0.25 gm., accurately weighed, in 1 mil of nitric acid, transfer to the cathode cup of an electrolytic apparatus,* which has previously been weighed with its metallic mercury, and dilute to about 30 mls. Electrolyze the solution, using a current of 1.5 to 2 amperes and 7 to 10 volts while rotating the anode about 500 revolutions per minute. When the electrolyte is free from mercury (to be ascertained by testing a few drops with hydrogen sulphide water), wash the cathode with water by siphonation, without interrupting the current, until the current drops to nearly zero. Remove the cup, wash the mercury with alcohol and then with ether, dry in a dessicator over sulphuric acid, weigh, and from the gain in weight calculate the amount of mercuric oxide. At least 99.5% of HgO should be found.

$$\text{Hg} \times 1.07976 = \text{HgO}.$$

Mercuric oxide may also be determined by volumetric means as follows: Dissolve about 0.5 gm. of the oxide, accurately weighed, in 10 mls of water and 5 mls of nitric acid, dilute the solution to 150 mls, add ferric ammonium sulphate solution and titrate with ammonium sulphocyanate tenth-normal solution.

$$1 \text{ mil of tenth-normal } \text{NH}_4\text{CNS} = 0.01083 \text{ gm. of HgO,} \\ \log 03463$$

* For a description of a suitable apparatus see Smith's *Electro-Analysis*, 5th ed., 63 (1911).

MERCURY OXIDE, YELLOW

(Yellow Mercuric Oxide)

HgO

Mol. Wt. 216.6

Yellow mercuric oxide is a light orange-yellow powder. It is almost insoluble in water, but is soluble in dilute nitric or hydrochloric acid, the solution in the latter acid being often slightly turbid. In dry condition it contains at least 99.5% of HgO. Like the red oxide, this yellow mercury oxide is darkened on exposure to light with the formation of mercurous oxide, and sometimes metallic mercury.

To distinguish between the yellow and the red oxides, otherwise than by the color, heat 0.5 gm. for two hours on a steam-bath with a solution of 1 gm. of oxalic acid in 10 mls of water and 1 mil of ammonia water, replacing from time to time the water lost by evaporation. The yellow oxide is thereby converted into white or yellowish-white mercuric oxalate, while the red oxide remains practically unchanged.

Yellow mercuric oxide is principally employed in reagents for coal-tar dyes, in the volumetric determination of hydrocyanic acid and in the detection of acetic acid in formic acid and of carbon monoxide in gas mixtures. It has also been proposed as a standard for preparing volumetric solutions for alkalimetry, iodometry, oxidimetry and argentometry. Regarding this use see Rosenthaler and Abelman, *Pharm. J.*, **91**, 144, 186 (1913); *abst.*, *Chem. Abst.*, **7**, 3726 (1913); Incze, *Z. anal. Chem.*, **56**, 177 (1917); *abst.*, *Chem. Abst.*, **11**, 2865 (1917).

Mercury oxide, yellow, should be stored in well-stoppered, amber-colored bottles and should be exposed to light as little as possible.

Maximum Limits of Impurities

Non-volatile Matter	0.1500%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0350%
Nitrates (N ₂ O ₄)	0.0160%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of the yellow mercuric oxide. Not more than 0.003 gm. of residue should remain.

Chlorides.—Dissolve 1 gm. in a mixture of 5 mls of nitric acid and 15 mls of water and add silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 1 gm. in a mixture of 5 mls of nitric acid and 20 mls of water, add barium nitrate solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Nitrates.—Dissolve 1 gm. in a mixture of 2 mls each of sulphuric acid and water and, when cold, overlay the solution with ferrous sulphate solution. No color should develop at the plane of contact of the liquids.

Quantitative Method.—The quantitative determination is carried out as described under Mercury Oxide, Red. At least 99.5% of HgO should be found.

MERCURY AND POTASSIUM IODIDE

(Mercuric Potassium Iodide)

HgI₂2KI

Mol. Wt. 786.48

Mercury and potassium iodide usually occurs as yellow, deliquescent, crystalline masses, readily soluble in water. The reagent is used principally for detecting alkaloids and albuminoids; in solutions for separating minerals; and for detecting alkali hydroxides in alkali carbonates. It should be stored in well-stoppered bottles.

Maximum Limit of Impurity

Insoluble Matter 0.0000%

Method of Testing

Insoluble Matter.—Dissolve 5 gm. in 10 mls of water. No insoluble residue should be left. Dilute the solution with 75 mls of water. It should remain clear.

METAPHENYLENEDIAMINE HYDROCHLORIDE**(Metadiaminobenzene Hydrochloride)**

Mol. Wt. 181.05

Metaphenylenediamine hydrochloride occurs usually as a white or slightly reddish-white, crystalline powder, easily soluble in water. Exposed to air and light the salt acquires a reddish color. Should the solution have this color it may be decolorized before use by warming it with previously ignited animal charcoal.

Metaphenylenediamine hydrochloride is used principally in 0.5% solution as a reagent for nitrous acid; also for the detection of aldehydes, ketones, hydrogen peroxide and ozone. It should be stored in well-stoppered bottles and protected from light.

Maximum Limit of Impurity

Inorganic Impurities 0.0500%

Method of Testing

Inorganic Impurities.—Ignite 1 gm. and weigh any residue that remains. The weight should not exceed 0.0005 gm.

METHYLENE BLUE**(Methylthionine Chloride)**

Mol. Wt. 373.82

Methylene blue is a dark green, crystalline powder or lustrous crystals. It is readily soluble in water or alcohol.

Methylene blue is used extensively in microscopy as a stain; in the analysis of milk for approximating its bacterial content; in urinalysis; in determining the putrescibility of waters and sewage effluents; as an indicator in iodometric titrations; and in the determination of chlorates, perchlorates, stannous chloride, titanous compounds, etc. For a discussion of these and other chemical uses of methy-

lene blue see J. Soc. Dyers & Colorists, 31, 183 (1915); *abst.*, Chem. Abst., 10, 25 (1916).

Maximum Limits of Impurities

Ash	1.0000%
Arsenic (As)	0.0075%
Zinc (Zn)	0.1500%
Matter Insoluble in Alcohol	1.0000%

Methods of Testing

Ash.—Ignite 1 gm. of methylene blue. Not more than 0.01 gm. of residue should remain.

Arsenic.—Intimately mix 0.2 gm. of the methylene blue with about 0.5 gm. each of potassium nitrate and anhydrous sodium carbonate and heat the powdered mixture until the organic matter is completely oxidized. Dissolve the residue in about 15 mls of 10% sulphuric acid and evaporate the solution over a flame until vapors of sulphuric acid begin to come off. Dilute the residue with 2 or 3 mls of water, mix it with 5 mls of stannous chloride solution and allow it to stand one hour. No dark color or precipitate should result.

Zinc.—Ignite 0.5 gm. at a temperature below a red heat until it is completely carbonized, boil the powdered residue with 10 mls of 10% hydrochloric acid for five minutes, filter, wash the residue with 10 mls of water, to the combined filtrate and washings add a few drops of nitric acid and boil the mixture. Add an excess of ammonia water, filter if necessary and to the filtrate add an equal volume of hydrogen sulphide water. The liquid should remain clear.

Matter Insoluble in Alcohol (Dextrin, etc.).—Dissolve 1 gm. in 50 mls of boiling alcohol, filter through a counterpoised filter, wash the residue on the filter with boiling alcohol until the washings are no longer blue, dry the residue at 100° C. and weigh it. Not more than 1% of insoluble matter should be found.

METHYL ORANGE

(Sodium Salt of Para-dimethyl-amino-azobenzene-sulphonic Acid)

$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{N}:\text{NC}_6\text{H}_4\text{SO}_3\text{Na}$. Mol. Wt. 327.27

Methyl orange is usually met with as an orange-yellow powder or crystalline scales. It is practically insoluble in alcohol and only slightly soluble in cold water; readily soluble in hot water, any excess of the salt separating from the solution on cooling.

The solution of 1.0 gm. in 1 liter of water is used as a stock indicator solution. One or two drops of this solution in a volume of 50 to 100 mls is usually sufficient for the best results; an excess of the indicator should be avoided. Many chemists have in fact had poor success in using this indicator, but have subsequently found they were using too much of it.

Methyl orange is useful in titrating most mineral acids, including arsenic and phosphoric acids, these two producing the end reaction at the instant of formation of the mono-basic arsenate and phosphate, respectively. For organic acids it is useless. It is suitable for the titration of strong bases, such as the fixed alkalis and ammonia, and is employed in water analysis for estimating the alkalinity of waters. It is practically unaffected by carbonic acid, one of its best characteristics, so that the titration of carbonates can be carried out in cold solution with ease and precision with its use.

Method of Testing

Sensitiveness.—To 100 mls of water add 1 drop of the 0.1% aqueous solution of methyl orange. The light yellow color of the liquid should be changed to red by the addition of 0.05 mil of tenth-normal hydrochloric acid, and on further addition of 0.05 mil of tenth-normal sodium hydroxide solution the original color should be restored.

METHYL RED

(Para-dimethyl-amino-azo-benzene-ortho-carboxylic Acid; Methyl Red Indicator)

$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{N}:\text{NC}_6\text{H}_4\text{COOH}$ Mol. Wt. 269.22

Methyl red usually occurs as a dark red powder or glistening violet crystals. It is almost insoluble in water, but is soluble in alcohol and in glacial acetic acid.

Ordinarily a solution containing 0.2 gm. in 100 mls of alcohol is employed as an indicator, one or two drops of this solution in about 100 mls of liquid being usually sufficient for obtaining a good end-point. It has been noted, however, that strongly alcoholic solutions when very old appear to lose in some degree their sensitiveness to alkalis, apparently due to ester formation. For this reason a 0.2% solution in dilute alcohol has been recommended as a stock solution. A saturated aqueous solution has also been proposed for the same purpose.

Methyl red in its application somewhat resembles methyl orange but is more sensitive to both hydrogen and hydroxyl ions. It is particularly useful in titrating ammonia, weak organic bases, and alkaloids. For the last-named purpose it is preferable to iodeosin in some cases, as for instance in titrating nicotine in tobacco extracts. It is generally unsuited to the titration of organic acids, but oxalic and picric acids are readily determined with its aid. Borates and cyanides are susceptible to estimation with methyl red indicator, but carbonic acid interferes with the sharpness of its end-point more than with methyl orange so that the latter is to be preferred in the presence of carbonates. Methyl red is useless as an indicator with sulphurous and phosphoric acids.

Method of Testing

Sensitiveness.—Mix 1 or 2 drops of the alcoholic 0.2% solution of methyl red with 100 mls of water which has been freed from carbon dioxide by long boiling in a plati-

num dish. The yellowish-red color of the mixture should be changed to yellow by the addition of 0.1 mil of hundredth-normal sodium hydroxide solution, and on further adding 0.2 mil of hundredth-normal hydrochloric acid solution the fluid should become red.

NESSLER'S SOLUTION

(Mercuric-Potassium Iodide Solution, Alkaline)

Nessler's solution is a clear, yellowish liquid, miscible with water and having a strongly alkaline reaction. It is usually prepared by bringing together solutions of potassium iodide and mercuric chloride and afterwards making the mixture strongly alkaline with caustic alkali. Formulas for its preparation are so numerous as to be found in nearly every work on chemical analysis and therefore no special reference is required here. The formulas often vary slightly from one another as to the quantities of the ingredients and the manipulations in preparing the solution. In most cases a slight excess of mercuric chloride is recommended which is said to increase the sensitiveness of the reagent. Its efficiency also increases on keeping.

Besides its extensive employment in the detection and determination of ammonia, it is also used to detect aldehydes, vinyl alcohol and hexamethylenamine.

Nessler's solution should be stored in tightly-closed, rubber-stoppered bottles and should at all times be protected from contact with even traces of ammonia vapor.

Method of Testing

Sensitiveness.—Add 2 mls of Nessler's solution to 50 mls of water containing 0.05 mgm. of NH_3 .^{*} A yellowish-brown color should be produced at once.

^{*}To measure conveniently this quantity of ammonia, dissolve 0.157 gm. of ammonium chloride in 1 liter of water. Each mil of this solution contains 0.05 mgm. of NH_3 .

PALLADIUM**Pd****At. Wt. 106.7**

Palladium for reagent use is generally in the form of palladium sponge or palladium black, the former a gray spongy mass and the latter a black powder. Both forms are readily soluble in nitric or nitrohydrochloric acid but only slowly soluble in hydrochloric acid.

Palladium has the power of absorbing large amounts of hydrogen and it is largely by virtue of this power that it finds use as a reagent. It is used chiefly in gas analysis in determining hydrogen and as a catalyser in hydrogenation reactions.

Maximum Limits of Impurities

Copper	0.0000%
Iron	0.0000%

Methods of Testing

Copper and Iron.—Dissolve the palladium in nitrohydrochloric acid and evaporate the excess of acid on the steam-bath. Dissolve the residue in water and add ammonia water until the flesh-colored precipitate of ammonium-palladous chloride redissolves. Then pass into the solution gaseous hydrochloric acid, whereby the palladium is precipitated as yellow palladosamine chloride. Filter and treat the filtrate with ammonia water in excess. No coloration or precipitation should ensue.

PALLADIUM CHLORIDE**(Palladous Chloride)****PdCl₂****Mol. Wt. 177.62**

Palladium chloride is usually obtained as a dark-brown powder, yielding a turbid solution in water in consequence of the formation of a basic salt; clearly soluble on adding hydrochloric acid. It contains about 60% of Pd (theory 60.08%). The salt is hygroscopic. From aqueous solutions of palladium chloride the salt, PdCl₂ + H₂O, may be

crystallized. This crystallized compound is not hygroscopic.

Palladium chloride is used chiefly in gas analysis as a reagent for carbon monoxide, ethylene and other gases; for the detection of mercury vapor in the air; as a reagent for cocaine; and for the detection and determination of iodine.

The salt should be stored in tightly-stoppered bottles.

Method of Testing

Quantitative Method.—Dissolve about 0.5 gm., accurately weighed, in 2 or 3 mls of hydrochloric acid and dilute to about 100 mls. Pass acetylene gas into the solution until the palladium is entirely precipitated. Filter, wash the precipitate free from chlorides, ignite it, cautiously at first, then more strongly, finally in a current of hydrogen, and weigh. About 60% of Pd should be found.

PARADIMETHYLAMINOBENZALDEHYDE

$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{CHO}$

Mol. Wt. 149.14

Paradimethylaminobenzaldehyde usually occurs as colorless to light yellow crystals, soluble in alcohol and in hot water; sparingly soluble in cold water. It melts at about 73° C.

The reagent is used chiefly for the detection of indol, skatol, pyrrhol, albumins, antipyrine, anthranilic acid and urobilinogen and in the diagnosis of scarlet fever. It should be stored in well-stoppered bottles.

Maximum Limit of Impurity

Inorganic Matter 0.0500%

Method of Testing

Inorganic Matter.—Ignite 1 gm. and weigh any residue that remains. The weight should not exceed 0.0005 gm.

PHENACETOLIN

Phenacetolin is a yellowish-brown powder, soluble in alcohol but only slightly soluble in water. Its constitution is not definitely known. It is obtained by the action of sulphuric acid on glacial acetic acid and phenol.

An indicator solution may be prepared by digesting 1 gm. with 100 mls of warm alcohol and filtering when cold. Two or 3 drops of this solution suffice for 100 mls of the fluid to be titrated.

Phenacetolin is particularly useful in titrating mixtures of alkali hydroxides and carbonates, the yellow color passing to red as the hydroxide is neutralized and again to yellow when the carbonate is fully decomposed by the acid solution used. Ammonia may be titrated using phenacetolin, and the indicator is sensitive to mineral acids but is of little use in determining organic acids. It is used for estimating alkaline carbonates in mixtures with other salts, such as the acetate, and in determining alkalinity in water analysis, for which purpose it is used like lacmoid.

Method of Testing

Sensitiveness.—Add 2 drops of the indicator solution, described above, to 100 mls of water and add 0.05 mil of tenth-normal sodium hydroxide solution. The light brown color of the liquid should change to pink. Now add 0.05 mil of tenth-normal hydrochloric acid solution. The color should change to golden yellow.

PHENOL

(Carbolic Acid)

C_6H_5OH

Mol. Wt. 94.08

Phenol occurs usually as colorless needle-shaped crystals or as a white crystalline mass, having a characteristic, somewhat aromatic odor. It is hygroscopic and dissolves in about 15 parts of water; it is easily soluble in alcohol, ether, glycerin or chloroform. When heated phenol melts, and

on cooling again congeals at a temperature not below 40° C. Excessive hydration or the presence of foreign substances (cresols) tends to lower the congealing point. Pure, anhydrous phenol congeals at 42.5°–43° C. and boils at 182.6° C. (corr.).

When exposed to the light phenol develops a red tint. It is also very sensitive to the action of iron, minute traces of which quickly discolor it.

Phenol when liquefied by warming may be kept in liquid condition by the addition of about 8% of water. This liquefied phenol is not miscible with more water until enough has been added to effect solution of the phenol present.

The reagent is used principally in the detection of aniline, nitrobenzene, albumin, glycerin, saccharin, lignin, rosin oil and lactic acid; as a standard in testing disinfectants; and in staining solutions employed in microscopy.

Phenol should be stored in well-stoppered bottles of blue or amber-colored glass and should be protected from light. It exerts a marked caustic action upon the skin and care should be exercised in handling it. Phenol vapor is inflammable and should therefore not be exposed to danger of ignition.

Maximum Limit of Impurity

Residue upon Evaporation 0.0500%

Method of Testing

Residue upon Evaporation.—Heat 10 gm. upon the steam-bath and weigh the non-volatile residue. The weight should not exceed 0.005 gm.

PHENOLPHTHALEIN

$C_{20}H_{14}O_4$

Mol. Wt. 318.21

Phenolphthalein is a white or yellowish-white, odorless powder. It is soluble in about 10 parts of alcohol, less readily in ether (about 50 parts) and is almost insoluble in water. It melts at about 257° C. (theory 253–4° C.).

A solution suitable for use as an indicator is made by dissolving 1 gm. of phenolphthalein in 100 mls of alcohol. This solution should be colorless. About 3 drops of the solution are usually sufficient for a volume of about 50 mls of the liquid to be titrated.

Phenolphthalein is a useful indicator in the titration of practically all mineral and organic acids. It works well in alcoholic liquids, thus allowing the titration of acids soluble in alcohol, such as benzoic, salicylic and the fatty acids. Boric acid may be titrated if the solution contains about 50% of glycerin, but not alone. Alkali and alkali earth hydroxides are easily estimated by its use. It is sensitive to carbonic acid, but carbonates and bicarbonates are readily titrated in boiling solution. It is useless for ammonia and the direct estimation of most organic bases, although fair results may be obtained with a few alkaloids. The acid radical of many alkaloidal salts may be titrated using phenolphthalein, the free base not affecting the indicator. Phenolphthalein is used in water analysis for differentiating carbonates and bicarbonates and in estimating free carbon dioxide by means of standard sodium carbonate solution; also in physiological chemistry to distinguish between transudates and exudates. Regarding the use of phenolphthalein for the detection of blood see Kastle, Chemical Tests for Blood, U. S. Public Health Service, Hygienic Laboratory, Bull. No. 51 (1909).

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Insoluble Matter	0.0000%
Fluorane	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of phenolphthalein. Any residue remaining should not weigh more than 0.001 gm.

Insoluble Matter.—Dissolve 1 gm. in 15 mls of alcohol. No insoluble matter should remain.

Fluorane.—Dissolve 0.5 gm. in 50 mls of 0.4% sodium hydroxide solution. Solution should be complete.

Sensitiveness.—To 250 mls of water, which has just been well boiled and then cooled, add 3 to 5 drops of phenolphthalein solution (1 gm. to 100 mls of alcohol). This mixture should require not more than 0.05 mil of tenth-normal sodium hydroxide solution to produce a red color.

PHENYLHYDRAZINE

$C_6H_5NHNH_2$

Mol. Wt. 108.11

Phenylhydrazine is a colorless or slightly yellowish, highly refractive liquid, boiling at about 241° to 243° C. (theory 243.5° C.), and solidifying when cooled, forming crystals which melt at about 19° C. (theory 19.35° C.). It is very slightly soluble in cold water, more readily in hot water, and is easily soluble in alcohol or ether.

Phenylhydrazine is a strong reducing agent. On exposure to air and light it soon acquires a dark color, due to oxidation. Its solutions also rapidly absorb oxygen from the air, becoming brown.

The reagent is used mainly in the determination of aluminum in the presence of iron; in the assay of benzaldehyde; as a reagent for aldehydes and ketones, fusel oil, wood fiber, molybdenum and for carbon disulphide in benzene and carbon tetrachloride; and in distinguishing between the di-oxybenzenes and between tannic and gallic acids.

Phenylhydrazine should be stored in tightly-closed, glass-stoppered bottles, well protected from light. It attacks the skin and its prolonged contact with the person should be avoided.

Maximum Limit of Impurity

Insoluble Matter 0.0000%

Method of Testing

Insoluble Matter.—Shake 2 mls of phenylhydrazine with 20 mls of 5% acetic acid. A clear solution should result.

PHENYLHYDRAZINE HYDROCHLORIDE

Mol. Wt. 144.58

Phenylhydrazine hydrochloride is usually in the form of white or slightly yellowish or brownish, crystalline scales. It is readily soluble in hot water and in alcohol. It melts at about 225° C. with decomposition. Like the base, phenylhydrazine hydrochloride darkens in color on exposure to air and light and becomes less soluble in water.

The reagent is used principally in the detection and differentiation of those sugars with which it forms characteristic, crystalline osazones and in the detection of formaldehyde, other aldehydes and urea.

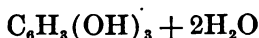
Phenylhydrazine hydrochloride should be stored in well-stoppered bottles, protected from light.

Maximum Limit of Impurity

Insoluble Matter 0.0000%

Method of Testing

Insoluble Matter.—Dissolve 2 gm. in 20 mls of warm water. The solution should be clear and free from suspended particles.

PHLOBOGLUCIN**(Phloroglucinol)**

Mol. Wt. 162.11

Phloroglucin is obtained as white or yellowish crystals or crystalline powder. It is soluble in about 95 parts of water and in about 10 parts of alcohol; readily soluble in ether. Its melting-point depends upon the method of determination, being about 218° C. (theory 217° to 219° C.) on rapid heating, but much lower on slow heating.

Phloroglucin is used principally as a reagent for wood fiber and for small quantities of free hydrochloric acid. For detecting the latter in gastric juice Günzburg's solution is much used. This consists of a solution of 2 parts of

phloroglucin and 1 part of vanillin in 30 parts of alcohol. Phloroglucin is also used in determining pentoses and pentosans; in detecting formaldehyde, methyl alcohol, peach-kernel oil and hydrated chloral; and in microscopy.

The reagent should be stored in well-stoppered bottles of amber glass.

Maximum Limit of Impurity

Diresorcin 0.0000%

Method of Testing

Diresorcin.—Heat to boiling a solution of 0.1 gm. of phloroglucin in 10 mls of acetic anhydride, cool the solution and superimpose it upon 10 mls of sulphuric acid. No violet color should appear at the plane of contact of the liquids.

PLATINUM

Pt

At. Wt. 195.2

Platinum for reagent use occurs in compact form as foil or wire and in a state of fine subdivision as platinum sponge and platinum black. Compact platinum has a silver-white color, platinum sponge is a grayish, spongy mass, while platinum black is a black powder. The metal is insoluble in nitric or hydrochloric acid but is soluble in nitrohydrochloric acid. The finely divided forms of platinum have great occlusive capacity and catalytic activity.

Platinum is used principally as a catalytic agent in various oxidation and reduction reactions; also for the preparation of platinum salts.

Maximum Limits of Impurities

Silver 0.0000%
Other Foreign Metals Traces

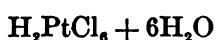
Methods of Testing

Silver.—Dissolve 0.5 gm. of the platinum in nitrohydrochloric acid, evaporate the solution to dryness and dissolve the residue in water. No white, insoluble portion should remain.

Other Foreign Metals.—Dissolve 1 gm. in nitrohydrochloric acid, evaporate the solution to dryness, strongly ignite the residue and digest the resulting spongy mass with 25 mls hot 10% nitric acid for fifteen minutes. Filter and evaporate the filtrate. Not more than traces of residue should remain.

PLATINUM CHLORIDE

(Platinic Chloride; Chlorplatinic Acid)



Mol. Wt. 518.07

Platinum chloride is a brownish-yellow, very deliquescent, crystalline mass, easily soluble in water and in alcohol, yielding solutions acid to litmus paper. It should contain about 37.5% of Pt.

Platinum chloride is chiefly employed in the detection and determination of potassium, rubidium, caesium, thallium and ammonium; as an alkaloidal reagent and as a catalyser.

The reagent should be stored in well-closed, glass-stoppered bottles.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Metals Soluble in Nitric Acid	0.2500%
Sulphates (SO_4)	0.0050%
Nitrates (N_2O_5)	0.0800%
Barium (Ba)	0.0020%

Methods of Testing

Insoluble Matter.—Dissolve 1 gm. in 10 mls of water and also 1 gm. in 10 mls of alcohol. Both solutions should be yellow in color and free from insoluble matter.

A 10% solution of pure platinic chloride has a yellow color; a red or brown color indicates the presence of platinous salt or of iridium.

Metals Soluble in Nitric Acid.—Strongly ignite 2 gm. and digest the residue with 25 mls of 10% nitric acid on a steam-bath for fifteen minutes. Filter, evaporate the fil-

trate and ignite and weigh the residue. The weight should not exceed 0.005 gm.

The slight residue nearly always found is due to substances derived from the vessels in which the platinum is dissolved and is difficult to avoid.

Sulphates.—Dissolve 1 gm. in 20 mls of water, add barium chloride solution and allow it to stand two to three hours. No precipitate should form.

Nitrates.—Mix 2 mls of the approximately 10% solution with 2 mls of sulphuric acid and overlay the mixture with 2 mls of ferrous sulphate solution. No brownish-red color should form at the plane of contact of the liquids.

Barium.—Dissolve 1 gm. in 20 mls of water, add a few drops of diluted sulphuric acid and allow it to stand two to three hours. No precipitate should form.

Quantitative Method.—Dissolve about 0.5 to 0.75 gm., accurately weighed, in 50 mls of water, add 10 mls of 27% solution of sodium hydroxide and 2 gm. of hydrated chloral, and warm the mixture on the steam-bath until the platinum is entirely reduced. Keep the reaction of the liquid strongly alkaline and add more hydrated chloral if necessary to complete the precipitation. Filter, wash and ignite the precipitate and weigh it as Pt. About 37.5% of Pt. should be found.

The salt may be assayed electrolytically as follows: Coat a platinum dish cathode with a rather thick layer of copper. Introduce an aqueous solution containing in each 25 mls about 0.25 gm. of the platinum chloride, accurately weighed, add 2.5 to 3 mls of 10% sulphuric acid and electrolyze with a current of 1 to 2 amperes, using a rapidly rotating disc anode. Twenty to thirty minutes are usually sufficient for complete deposition. Wash the deposit with water and alcohol, dry and weigh it.

The platinum content may be approximated by carefully igniting the platinum chloride and weighing the residue as Pt.

POTASSIUM ACETATE**KC₂H₃O₂****Mol. Wt. 96.13**

Potassium acetate is usually in the form of colorless, lustrous crystals, soluble in about 0.5 part of water and in about 3 parts of alcohol. Its solutions have an alkaline reaction to litmus. The salt is very deliquescent. Upon ignition it gives off inflammable vapors and is converted to potassium carbonate. The titration of this residue affords a convenient method of assaying the salt.

The reagent is chiefly used in the detection of tartaric acid and of alcohol in oils and ethers. It should be stored in well-stoppered bottles and exposed to the air as little as possible.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0050%
Calcium (Ca)	0.0100%
Heavy Metals	0.0000%

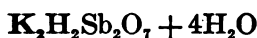
Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add a few mls of nitric acid and of silver nitrate solution. Not more than a slight opalescence should result.

Sulphates.—Dissolve 2 gm. in 20 mls of water and add a few drops of hydrochloric acid and some barium chloride solution. No change should be apparent.

Calcium.—Dissolve 2 gm. in 20 mls of water and add a few drops of ammonia water and some ammonium oxalate solution. The liquid should not appear changed.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water and add a few drops of hydrochloric acid and some hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should result.

POTASSIUM ANTIMONATE**(Potassium Acid Pyroantimonate)**

Mol. Wt. 504.68

Potassium antimonate occurs usually as a white, granular, crystalline powder soluble in about 35 parts of water, more readily in boiling water.

The reagent is used principally for the detection of sodium. For this purpose a solution is prepared just before use by dissolving 1 gm. in 100 mls of boiling water and filtering. The solution to be tested for sodium should be neutral or slightly alkaline.

Methods of Testing

Suitability as a Reagent for Sodium.—Dissolve 1 gm. in 100 mls of boiling water and filter if necessary. The solution should be neutral to litmus paper.

Add 20 mil portions of this solution of potassium antimonate to a solution of 1 gm. of potassium chloride in 10 mls of water and to a solution of 1 gm. of ammonium chloride in 10 mls of water and allow the mixtures to stand fifteen minutes. No precipitate should form in either. A precipitate occurring in either test indicates that the potassium antimonate is unsuitable for use as a reagent for sodium.

POTASSIUM BICARBONATE

Mol. Wt. 100.11

Potassium bicarbonate usually occurs as colorless, transparent crystals, soluble in about 4 parts of water, almost insoluble in alcohol. Its solutions are slightly alkaline to litmus. The salt contains at least 99% of KHCO_3 .

In closed containers the salt is stable but in a stream of air, or of nitrogen or hydrogen, carbon dioxide is very gradually lost and potassium carbonate remains. On heating this change also takes place, at high temperatures very

rapidly; in solutions of the salt carbon dioxide is lost, slowly when cold, very quickly on heating.

Potassium bicarbonate is used chiefly as a neutralizing agent, especially preceding titrations with iodine solution, as in determining arsenous and antimonous compounds; as a source of carbon dioxide in nitrogen determinations by combustion; and in the separation of alkaloids.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0040%
Calcium (Ca)	0.0010%
Aluminum (Al)	0.0400%
Silicates	0.0000%
Phosphates (P_2O_5)	0.0040%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0030%
Nitrates (N_2O_5)	0.0011%

Methods of Testing

Sulphates.—Dissolve 3 gm. of potassium bicarbonate in 50 mls of water, add 5 mls of hydrochloric acid, boil the solution for several minutes, add barium chloride solution and allow it to stand at least twelve hours. No precipitate should form.

Chlorides.—Dissolve 3 gm. in 50 mls of water and add 10 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should develop.

Nitrates.—Dissolve 3 gm. of potassium bicarbonate and a few milligrams of sodium chloride in 15 mls of 10% sulphuric acid, add 1 drop of indigo solution and 10 mls of concentrated sulphuric acid and shake the mixture. The blue color should persist after mixing.

Silicates.—Dissolve 5 gm. in a mixture of 20 mls of water and 15 mls of hydrochloric acid. Evaporate the solution in a platinum dish, dry the residue at 120°C . and dissolve it in 25 mls of water acidulated with 2 to 3 mls of hydrochloric acid. The solution should be perfectly clear.

Phosphates.—Dissolve 5 gm. in 50 mls of water, add 40 mls of nitric acid and 25 mls of ammonium molybdate solution and allow it to stand two hours at about 40° C. No yellow precipitate should form.

Calcium and Aluminum.—Dissolve 5 gm. in a mixture of 25 mls of water and 15 mls of 36% acetic acid, add a slight excess of ammonia water and heat the mixture on the steam-bath for half an hour. No flocculent precipitate should form. Then add a few mls of ammonia water and some ammonium oxalate solution. No change should be apparent.

Heavy Metals.—Dissolve 2 gm. in 10 mls of water, add 3 mls of hydrochloric acid and 5 to 10 mls of hydrogen sulphide water. No change should be apparent. Slightly supersaturate the mixture with ammonia water and add a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Quantitative Method.—Dry the salt over sulphuric acid, weigh out a sample of the dried material, dissolve it in water and titrate with standard acid solution, using methyl orange as indicator. At least 99% of KHCO_3 should be found.

1 mil of normal HCl = 0.10011 gm. of KHCO_3 , log 00047.

POTASSIUM BINIODATE

KIO_3HIO_3

Mol. Wt. 389.95

Potassium biniodate usually occurs as small, white crystals, clearly and completely soluble in 75 parts of water but insoluble in alcohol. Its solutions are acid to litmus. The salt contains at least 99.95% of KIO_3HIO_3 .

The reagent is used chiefly as a standard for preparing volumetric solutions for both iodometry and acidimetry. For this purpose it should be dried to constant weight at 95 to 98° C. A temperature much above 100° C. is likely to decompose the salt.

Methods of Testing

Quantitative Method.—The determination is to be made both iodometrically and acidimetrically. At least 99.95% of KIO_3HIO_3 should be found in each case.

Iodometric: Dry the salt to constant weight at 95° to 98° C., weigh out 0.10 to 0.15 gm. of the dried material, dissolve it in 20 mls of water, add 3 gm. of potassium iodide and 5 mls of hydrochloric acid, dilute the liquid with 100 mls of water and titrate the liberated iodine with tenth-normal sodium thiosulphate solution.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.00324958$ gm. of KIO_3HIO_3 , log 51182.

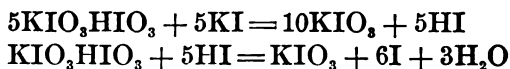
Acidimetric: Dry the salt to constant weight at 95° to 98° C., weigh out accurately about 1.5 gm. of the dried material dissolve it in 100 mls of water and titrate it in boiling solution with tenth-normal sodium hydroxide solution, using phenolphthalein as indicator.

1 mil of tenth-normal $\text{NaOH} = 0.038995$ gm. of KIO_3HIO_3 , log 59101.

The reaction occurring when potassium biniodate is treated with potassium iodide in acid solution, as in the iodometric method above, is represented by the following equation in which one molecule of the biniodate liberates 12 atoms of iodine:



In the absence of an acid the reaction is



each molecule of KIO_3HIO_3 liberating one atom of iodine.

To perform the titration without the addition of an acid add 3 gm. of perfectly neutral potassium iodide to the solution of about 1 gm. of the biniodate, accurately weighed, in 20 mls of water, dilute to 100 mls and titrate with tenth-normal sodium thiosulphate solution. The water used should be free from carbon dioxide.

This titration may be combined with the one made in the presence of an acid for proving the correct composition of the biniodate under examination. Exactly twelve times as much iodine should be liberated in the latter as in the former if the salt is pure KIO_3HIO_3 . It is obvious also that this comparison could be made with the use of a thio-sulphate solution of unknown strength. The method, therefore, has special value in not depending upon the standardization of the thiosulphate solution by other means.

POTASSIUM BISULPHATE

(Potassium Acid Sulphate)

KHSO_4

Mol. Wt. 136.16

Potassium bisulphate is usually in the form of colorless crystals, soluble in 3 parts of water and yielding a solution acid to litmus. Moderate heat does not affect the salt, but under ignition it loses sulphuric acid and is transformed to the normal potassium sulphate. It contains usually about 99% to 100% of KHSO_4 .

The reagent is used principally as a flux in the analysis of ores and siliceous compounds; in the determination of radium; and in the detection of artificial coloring in wine.

Maximum Limits of Impurities

Heavy Metals	0.0000%
Chlorides (Cl)	0.0050%
Arsenic (As)	0.0015%

Methods of Testing

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add silver nitrate solution. Not more than a slight opalescence should be produced.

Arsenic.—Powder 1 gm. of the salt, mix it with 3 to 4 mils of stannous chloride solution and allow it to stand one hour. No dark color should develop.

Quantitative Method.—Dissolve the salt in water and titrate the solution with a standard alkali solution, using methyl orange as indicator.

1 mil of normal NaOH = 0.13618 gm. of KHSO_4 ,
log 13411.

POTASSIUM BISULPHITE, META

(Potassium Pyrosulphite)

$\text{K}_2\text{S}_2\text{O}_5$

Mol. Wt. 222.32

Potassium meta-bisulphite is usually in the form of a white, crystalline powder, easily soluble in water. Its solutions are acid to litmus. The reagent contains at least 95% of $\text{K}_2\text{S}_2\text{O}_5$.

Potassium meta-bisulphite possesses a slight odor of sulphur dioxide which is more noticeable when the salt is rubbed. Exposed to the air the salt gradually oxidizes to potassium sulphate. This change takes place more readily in solution or on heating.

The reagent is used principally as a reducing agent and also in photography. It should be stored in tightly-stoppered bottles in a cool place.

Maximum Limits of Impurities

Chlorides (Cl)	0.0100%
Heavy Metals	Trace
Arsenic (As)	0.0005%

Methods of Testing

Chlorides.—Dissolve 0.5 gm. in 25 mils of water, add 1 gm. of sodium peroxide and evaporate the solution to dryness. Dissolve the residue in 20 mils of water, acidulate with nitric acid and add silver nitrate solution. Not more than a slight opalescence should result.

Heavy Metals.—Dissolve 1 gm. in 10 mls of water, add 10 mls of hydrochloric acid, evaporate on the steam-bath to dryness, dissolve the residue in 20 mls of water, acidulate with a few drops of hydrochloric acid and add hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No precipitate and not more than a slight green color should be produced.

Arsenic.—Add 2 gm., a little at a time, to 10 mls of nitric acid, add 3 or 4 mls of sulphuric acid, evaporate the mixture on a sand-bath until vapors of sulphuric acid begin to come off, dissolve the residue in 25 mls of water, introduce the solution into a Marsh apparatus and allow the action to proceed one hour. No deposit of arsenic should form in the reduction tube.

Quantitative Method.—Dissolve about 0.15 to 0.2 gm., accurately weighed, in 50 mls of tenth-normal iodine solution, acidulate with 5 mls of hydrochloric acid and titrate the excess iodine with tenth-normal sodium thiosulphate solution. At least 95% of $K_2S_2O_5$ should be found.

1 mil of tenth-normal I = 0.005585 gm. of $K_2S_2O_5$,
log 74702.

POTASSIUM BITARTRATE

(Potassium Acid Tartrate)

$KHC_4H_4O_6$

Mol. Wt. 188.16

Potassium bitartrate usually occurs as a white, crystalline powder. It is soluble in about 200 parts of water and insoluble in alcohol; it dissolves in about 20 parts of boiling water. The reagent contains at least 99.9% of $KHC_4H_4O_6$. The salt dissolves readily in solutions of the alkalis or alkali carbonates with formation of the normal potassium tartrate. It is also readily soluble in mineral acids with transposition. The salt may be dried at 100° to 120° C. without injury.

The chief use of the reagent is for the standardization of volumetric solutions of the alkalis, which is performed in hot solution, using phenolphthalein as indicator.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Moisture	0.0000%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0200%
Ammonium Compounds (NH ₄)	0.0020%
Calcium (Ca)	0.0250%
Heavy Metals	0.0000%

Methods of Testing

Moisture.—Dry 5 gm. of the potassium bitartrate at 100° C. No loss in weight should result.

Chlorides.—Dissolve 1 gm. in 20 mls of water with the aid of 5 mls of nitric acid and add silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 1 gm. in 20 mls of water with the aid of 1 ml of nitric acid, add barium nitrate solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Ammonium Compounds.—Heat 2 gm. with 10 mls of 10% sodium hydroxide solution and test the vapors with moist red litmus paper. The paper should not turn blue.

Calcium.—Dissolve 1 gm. in 25 mls of boiling water acidulated with 5 mls of 36% acetic acid, allow it to cool thoroughly, filter, to the filtrate add several drops of ammonium oxalate solution and allow it to stand twelve to eighteen hours. The mixture should show no turbidity.

Heavy Metals.—Dissolve 5 gm. in a mixture of 25 mls of ammonia water and 25 mls of water and add hydrogen sulphide water. No change should be apparent.

Quantitative Method.—Dissolve about 5 gm., accurately weighed, in 250 mls of hot water, heat the liquid to boiling and titrate with normal alkali solution, using phe-

nolphthalein as indicator. At least 99.9% of $\text{KHC}_4\text{H}_4\text{O}_6$ should be found.

1 mil of normal $\text{NaOH} = 0.18816$ gm. of $\text{KHC}_4\text{H}_4\text{O}_6$,
log 27453.

Check the determination by cautiously igniting about 3 gm., accurately weighed, dissolving the residue in water and titrating with normal acid solution, using methyl orange as indicator.

1 mil of normal $\text{HCl} = 0.18816$ gm. of $\text{KHC}_4\text{H}_4\text{O}_6$,
log 27453.

POTASSIUM BROMATE

KBrO_3

Mol. Wt. 167.02

Potassium bromate occurs as white crystals or a crystalline powder, soluble in about 15 parts of water but only very slightly soluble in alcohol. The aqueous solution is neutral to litmus. The salt contains at least 99.9% of KBrO_3 .

Potassium bromate may be heated to 150°C . without change, but when heated to higher temperatures it decrepitate, melts and is converted to bromide with loss of oxygen.

The reagent is used chiefly as an oxidizing agent, especially in the determination of phenol, sulphurous acid, hydrogen sulphide, sulphur dioxide and oxalic acid; as a standard in iodometry and as a constituent of Koppeschaar's volumetric bromine solution.

Maximum Limit of Impurity

Potassium Bromide (KBr) 0.0400%

Methods of Testing

Potassium Bromide.—Dissolve 2 gm. in 30 mls of water and add a few drops of diluted sulphuric acid. The solution should not at once acquire a yellow color.

Quantitative Method.—Dry the salt over sulphuric acid to constant weight, weigh out accurately about 0.1 to 0.15

gm. of the dried salt, dissolve it in 20 mls of water, add 3 gm. of potassium iodide and 5 mls of hydrochloric acid and titrate the liberated iodine with tenth-normal solution of sodium thiosulphate. At least 99.9% of KBrO_3 should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.00278367$ gm. of KBrO_3 ,
log 44462.

POTASSIUM BROMIDE

KBr

Mol. Wt. 119.02

Potassium bromide is usually in the form of colorless or white crystals or granules. It is soluble in about 2 parts of water and in about 200 parts of alcohol, forming neutral or faintly alkaline solutions. The salt contains at least 98.5% of KBr and not more than 1.5% of KCl.

Potassium bromide is used chiefly in qualitative and quantitative reagents for urea; in detecting small quantities of copper; and in preparing Koppeschaar's standard bromine solution for estimation of phenol.

Maximum Limits of Impurities

Free Alkali (as K_2CO_3)	0.0346%
Bromates (Br_2O_3)	0.0025%
Iodides (I)	0.1000%
Sulphates (SO_4)	0.0100%
Barium (Ba)	0.0020%
Heavy Metals	0.0000%
Potassium Chloride (KCl)	1.5000%

Methods of Testing

Free Alkali.—Dissolve 1 gm. in 20 mls of water and add 0.05 mil of tenth-normal sulphuric acid solution and a few drops of phenolphthalein solution. No red color should result.

Bromates.—Dissolve 1 gm. in 10 mls of water, add a few drops of 10% sulphuric acid and a little chloroform and shake the mixture. The chloroform should not become yellow.

Iodides.—Dissolve 1 gm. in 20 mls of water, add a few drops of ferric chloride solution and some starch solution and allow it to stand ten minutes. No blue color should develop.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add a few drops of hydrochloric acid and some barium chloride solution. No turbidity should result.

Barium.—Dissolve 1 gm. in 20 mls of water and add a few drops of 10% sulphuric acid. No turbidity should result.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent.

Quantitative Method and Test for Potassium Chloride.—Dry the salt at 100° C., weigh accurately 0.3 to 0.4 gm. of the dried material, dissolve it in water and titrate with tenth-normal solution of silver nitrate, using potassium chromate as indicator. At least 98.5% of KBr should be found.

1 mil of tenth-normal $\text{AgNO}_3 = 0.011902$ gm. of KBr,
log 07562.

Each gram of the dried potassium bromide should require not more than 84.77 mls of the tenth-normal silver nitrate solution.

POTASSIUM CARBONATE

K_2CO_3

Mol. Wt. 138.3

Potassium carbonate is usually in the form of a white, granular powder. It is soluble in about 1 part of water but is practically insoluble in alcohol. It is very hygroscopic and deliquescent. The salt contains at least 99% of K_2CO_3 .

Potassium carbonate vaporizes to some extent in the hottest part of a Bunsen flame, and at lower temperatures loses some carbon dioxide which it again takes up from the air on cooling.

The reagent is used principally as a neutralizing agent; as a constituent of fluxes for the decomposition of silicates and various insoluble salts; for the preparation of potassium salts; and as a dehydrating agent.

The salt should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Heavy Metals	0.0000%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0050%
Nitrates (N ₂ O ₅)	0.0800%
Phosphates (P ₂ O ₅)	0.0040%
Silicates	0.0000%
Sulphides (S)	0.0200%
Sulphites (SO ₃)	0.0080%
Cyanides (CN)	0.0480%
Aluminum (Al)	0.0400%
Calcium (Ca)	0.0010%

Methods of Testing

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water; to another solution of 1 gm. in 20 mls of water add 36% acetic acid in slight excess and then add hydrogen sulphide water. No change should be apparent in either case.

Chlorides.—Dissolve 1 gm. in 20 mls of water, acidify with nitric acid and add silver nitrate solution. Not more than a slight opalescence should develop.

Sulphates.—Dissolve 1 gm. in 20 mls of water, acidify slightly with hydrochloric acid, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Nitrates.—Dissolve 0.2 gm. in 2 mls of 10% sulphuric acid, mix the solution with 2 mls of concentrated sulphuric acid and overlay the mixture with 1 ml of ferrous sulphate solution. No brown color should develop at the plane of contact of the liquids.

Phosphates.—Dissolve 5 gm. in 50 mls of water, add 50 mls of nitric acid and 25 mls of ammonium molybdate

solution and allow it to stand two hours at about 40° C. No yellow precipitate should form.

Silicates.—Dissolve 5 gm. in 20 mls of water and 20 mls of hydrochloric acid in a platinum dish, evaporate the solution to dryness, dry the residue half an hour at about 120° C. and then dissolve it in a mixture of 3 mls of hydrochloric acid and 25 mls of water. The solution should be perfectly clear.

Sulphides and Sulphites.—Dissolve 1 gm. in 20 mls of water, pour 1 mil of this solution into 10 mls of tenth-normal solution of silver nitrate and heat the mixture to 60° to 70° C. The yellowish-white precipitate first formed should not be rendered darker by the heating.

Cyanides.—Dissolve 1 gm. in 20 mls of water, mix 5 mls of this solution with a solution of 0.5 gm. of ferrous sulphate in 5 mls of water, add 1 or 2 drops of ferric chloride solution, heat to about 70° C. and acidulate with hydrochloric acid. No green color or blue precipitate should be produced.

Aluminum and Calcium.—Dissolve 5 gm. in 25 mls of water, add 25 mls of 36% acetic acid followed by 15 mls of ammonia water and heat it half an hour on the steam-bath. No flocculent precipitate should form. Then add ammonium oxalate solution to the liquid. No change should be apparent.

Quantitative Method.—Dissolve the salt in water and titrate the solution with standard acid solution, using methyl orange as indicator. At least 99% of K_2CO_3 should be found.

1 mil of normal HCl = 0.0691 gm. of K_2CO_3 , log 83948.

POTASSIUM CHLORATE

$KClO_3$

Mol. Wt. 122.56

Potassium chlorate is usually in the form of colorless, lustrous crystals or granules, soluble in about 17 parts of

water but almost insoluble in alcohol or in ether. Its aqueous solution is neutral to litmus. The salt contains at least 99% of KClO_3 .

Potassium chlorate is a strong oxidizing agent. It readily gives up its oxygen on heating or in the presence of reducing substances. Dangerous explosions are liable to occur when the material is heated, subjected to concussion or pressure or triturated with organic matter, such as sugar, tannin, cork, dust, etc., or with sulphur, sulphides, or other easily oxidizable substances.

In the presence of hydrochloric acid chlorine is liberated; extemporaneous preparation of chlorine water may thus be accomplished if the presence of potassium salts is not objectionable. Warmed with sulphuric acid the salt decomposes with characteristic loud decrepitation.

The reagent is used chiefly as an oxidizer; in forensic investigations; in the determination of sulphur; as a reagent for alkaloids, phenol and indican; and in the Parr calorimeter.

Potassium chlorate should be stored in well-stoppered bottles in a cool place, and should be handled with caution.

Maximum Limits of Impurities

Chlorides (Cl)	0.0010%
Heavy Metals	0.0000%
Calcium (Ca)	0.0200%
Bromates (Br_2O_3)	0.0800%
Nitrates (N_2O_5)	0.0016%
Sulphates (SO_3)	0.0030%
Arsenic (As)	0.0002%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add silver nitrate solution. No change should be apparent.

Heavy Metals.—Dissolve 2 gm. in 25 mls of water and add hydrogen sulphide water. The liquid should not appear changed.

Calcium.—Dissolve 1 gm. in 20 mls of water and add a few drops of ammonia water and some ammonium oxalate solution. No change should be apparent.

Bromates.—Ignite gently 1 gm. of potassium chlorate, dissolve the residue in water, add a few drops of chlorine water and shake the mixture with a little chloroform. The latter should not become colored yellow or brown.

Nitrates.—Heat 1 gm. with 5 mls of 27% sodium hydroxide solution and 0.5 gm. each of zinc dust and reduced iron (or 1 gm. of Devarda's metal) and test the vapors for ammonia by means of moistened red litmus paper. No ammonia should be evolved.

Sulphates.—Dissolve 1 gm. of the potassium chlorate in 20 mls of water, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form in this time.

Arsenic.—Pour 40 mls of hydrochloric acid over 5 gm. of potassium chlorate in a porcelain dish. As soon as the evolution of chlorine has slackened, heat the mixture on a steam-bath until free from the odor of chlorine, cool it, dilute with water, transfer it a little at a time to the generating flask of a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

Quantitative Method.—Dissolve about 0.1 gm., accurately weighed, in 10 mls of water in a flask of about 250 mls capacity. Add 25 mls of acid ferrous sulphate solution (see page ix), insert a stopper provided with a Bunsen valve and boil the mixture for ten minutes. Cool the mixture, add 10 mls of acid manganese sulphate solution (see page ix) and titrate the excess of ferrous sulphate with tenth-normal potassium permanganate solution. At the same time titrate another portion of 25 mls of the acid ferrous sulphate solution. From the difference between the results obtained in the two titrations at least 99% of KClO_3 should be found.

1 mil of tenth-normal $\text{KMnO}_4 = 0.0020427$ gm. of KClO_3 ,
log 31021.

POTASSIUM CHLORIDE**KCl****Mol. Wt. 74.56**

Potassium chloride is usually in the form of colorless crystals, or a white crystalline powder, soluble in about 3 parts of water; it is insoluble in absolute alcohol or ether. The aqueous solution is neutral to litmus.

The reagent finds its principal uses in the detection and determination of silicofluorides and in photography.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0030%
Nitrates (N_2O_5)	0.0032%
Chlorates (Cl_2O_5)	0.0180%
Heavy Metals	0.0000%
Calcium (Ca)	0.0100%
Magnesium (Mg)	0.0020%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 20 mls of water, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add 1 drop of indigo solution and 10 mls of sulphuric acid and shake the mixture. The blue color should persist after shaking.

Chlorates.—Dissolve 1 gm. in 20 mls of water and add zinc iodide-starch solution and a little hydrochloric acid. No blue color should result.

Heavy Metals.—Dissolve 3 gm. in 50 mls of water and add a few drops of ammonia water and ammonium sulphide solution. No dark color or precipitate should result.

Calcium.—Dissolve 3 gm. in 50 mls of water and add a few drops of ammonia water and some ammonium oxalate solution. No change should be apparent.

Magnesium.—Dissolve 1 gm. in 5 mls of water, add 5 mls of ammonia water and some ammonium phosphate solution and allow it to stand twelve to eighteen hours. No precipitate should form.

POTASSIUM CHROMATE**(Yellow Potassium Chromate)** K_2CrO_4

Mol. Wt. 194.2

Potassium chromate usually occurs as lemon-yellow crystals, soluble in about 2 parts of water but insoluble in alcohol. Its aqueous solutions are alkaline to litmus. The salt contains at least 99.5% of K_2CrO_4 .

Potassium chromate is used chiefly in the detection of silver, lead, barium and mercury; in the determination of lead and barium; and as an indicator in argentometry.

Maximum Limits of Impurities

Free Alkali (as KOH)	0.2000%
Sulphates (SO_4)	0.0500%
Chlorides (Cl)	0.0050%
Aluminum (Al)	0.0250%
Calcium (Ca)	0.0050%

Methods of Testing

Free Alkali.—Dissolve 0.1 gm. in 25 mils of water and add phenolphthalein solution. No red color should be produced.

Sulphates.—Dissolve 2.5 gm. in 100 mils of water, add 25 mils of hydrochloric acid and some barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate of barium sulphate should form. Insufficient hydrochloric acid allows the precipitation of barium chromate.

Chlorides.—Dissolve 1 gm. in 20 mils of water, add 10 mils of nitric acid, warm the mixture to about 50° C., add a few drops of silver nitrate solution and allow it to stand 5 minutes. No turbidity should result.

Aluminum.—Dissolve 2 gm. in 35 mils of water, add 5 mils of ammonia water, heat the mixture on the steam-bath until the odor of ammonia has almost disappeared and allow it to stand twelve to eighteen hours. No precipitate should form.

Calcium.—Dissolve 2 gm. in 30 mls of water, add 5 mls of ammonia water and some ammonium oxalate solution and allow it to stand two hours. No precipitate should form.

Quantitative Method.—Dissolve 0.1 to 0.2 gm., accurately weighed, in 10 mls of water, add 2 to 3 gm. of potassium iodide followed by 10 mls of 10% sulphuric acid and dilute with about 350 mls of water which has been thoroughly boiled and then cooled. Titrate the liberated iodine with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 99.5% of K_2CrO_4 should be found.

1 ml of tenth-normal $Na_2S_2O_3 = 0.0064733$ gm. of K_2CrO_4 , log 81113.

POTASSIUM DICHROMATE

(Potassium Bichromate)

$K_2Cr_2O_7$

Mol. Wt. 294.2

Potassium dichromate generally occurs as dark, yellowish-red crystals or granules. It is soluble in about 10 parts of water, the solution having an acid reaction to litmus. It contains at least 99.9% of $K_2Cr_2O_7$.

Potassium dichromate is chiefly used in separating the alkali earth metals; in standardizing solutions of sodium thiosulphate and in preparing standard dichromate solutions used in oxidimetry; in the evaluation of zinc dust and the quantitative determination of alcohol, aldehydes, glycerin, copper, uranium, bismuth, nitrogen, hydrogen, sulphur and bromine; as a reagent for a large number of substances including many alkaloids and glucosides, sugars, alcohols, aliphatic hydroxy-acids, unsaturated fatty acids, aniline and anilides, benzidine, alphanaphthol, phenol, antipyrine, cantharidin, picrotoxin and cottonseed oil; and as a hardening agent in microscopy.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0250%
Chlorides (Cl)	0.0050%
Aluminum (Al)	0.0250%
Calcium (Ca)	0.0050%

Methods of Testing

Sulphates.—Dissolve 5 gm. in 100 mls of water, add 30 mls of hydrochloric acid and some barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form. Absence or insufficiency of hydrochloric acid permits the precipitation of barium chromate which obscures the sulphate reaction.

Chlorides.—Dissolve 1 gm. in 20 mls of water, add 10 mls of nitric acid, heat the mixture to about 50°C ., add a few drops of silver nitrate and allow it to stand ten minutes. No turbidity should develop.

Aluminum.—Dissolve 2 gm. in 35 mls of water, add 5 mls of ammonia water, heat the solution on the steam-bath until the odor of ammonia has almost disappeared and allow it to stand at ordinary temperature for twelve to eighteen hours. No precipitate should form.

Calcium.—Dissolve 2 gm. in 30 mls of water, add 5 mls of ammonia water and some ammonium oxalate solution and allow to stand two to three hours. No precipitate should form.

Quantitative Method.—Dissolve 0.1 to 0.2 gm., accurately weighed, in 25 mls of water which has just been thoroughly boiled and then cooled. Add 2 gm. of potassium iodide and 10 mls of 10% sulphuric acid, then dilute the mixture with about 350 mls of the boiled water and titrate the liberated iodine with tenth-normal solution of sodium thiosulphate. At least 99.9% of $\text{K}_2\text{Cr}_2\text{O}_7$ should be found.
 1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.0049033$ gm. of $\text{K}_2\text{Cr}_2\text{O}_7$,
 log 69049.

POTASSIUM FERRICYANIDE**(Red Prussiate of Potash)** **$K_3Fe(CN)_6$** **Mol. Wt. 329.23**

Potassium ferricyanide is usually obtained as bright, ruby-red crystals, soluble in about 2.5 parts of water. Its 5% aqueous solution is neutral. It contains at least 99% $K_3Fe(CN)_6$. The salt is sensitive to light and on exposure to it is gradually converted into the ferrocyanide. This change is also readily effected by other reducing agents.

Potassium ferricyanide is used chiefly in detecting ferrous salts, nitric acid, hydrogen peroxide, cobalt, pyrogallol, morphine, strychnine, caffeine and reducing agents generally; in differentiating alpha and beta naphthol; in estimating saccharose and dextrose; and as an indicator in the dichromate titration of ferrous salts.

The salt should be stored in well-stoppered bottles of amber-colored glass. Its solutions should be freshly prepared when required.

Maximum Limits of Impurities

Ferrous Salts (Fe^{++})	0.0558%
Sulphates (SO_4)	0.0050%
Chlorides (Cl)	0.0100%

Methods of Testing

Ferrous Salts.—Dissolve 2 gm. in 400 mls of water, add 10 mls of diluted sulphuric acid and titrate the solution with tenth-normal potassium permanganate solution. At most 0.2 ml of the permanganate solution should be necessary to effect a color change from yellowish-green to yellowish-red. In order better to observe the color change compare the color of the solution titrated with that of another composed of the same amounts of ferricyanide, water and sulphuric acid.

Sulphates.—Dissolve 1 gm. in 20 mls of water, add 1 ml of hydrochloric acid and some barium chloride solution and allow the mixture to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

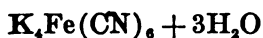
Chlorides.—Deflagrate a mixture of 0.5 gm. of the powdered salt and 1 gm. of potassium nitrate (tested for chlorides and perchlorates as directed under Potassium Nitrate, page 274) by introducing small quantities at a time into a porcelain crucible heated to redness. Ignite for a few minutes and leach the melt with 20 mls of water. Filter, add to the filtrate 0.5 gm. of potassium nitrate, evaporate to dryness, refuse in a porcelain crucible until quiescent and allow to cool. When cold, dissolve in 20 mls of water, add 2 mls of nitric acid and some silver nitrate solution. Not more than an opalescence should be produced.

Quantitative Method.—Dissolve about 0.7 gm., accurately weighed, in 50 mls of water, add 3 gm. of potassium iodide and 1.5 gm. of zinc sulphate (meeting the test for iron under Zinc Sulphate, page 369), mix thoroughly, and titrate the liberated iodine with tenth-normal sodium thio-sulphate solution, using starch solution as indicator. At least 99% of $K_3Fe(CN)_6$ should be found.

1 mil of tenth-normal $Na_2S_2O_3 = 0.03292$ gm. of $K_3Fe(CN)_6$, log 51750.

POTASSIUM FERROCYANIDE

(Yellow Prussiate of Potash)



Mol. Wt. 422.38

Potassium ferrocyanide is usually in the form of yellow crystals, soluble in about 4 parts of water; insoluble in alcohol. The 5% solution is neutral to litmus. It contains at least 99% of $K_4Fe(CN)_6 + 3H_2O$.

Potassium ferrocyanide is used mainly in the detection of copper, iron, uranium and some other metals with which it forms characteristic compounds; for the detection of albumin, glucose, nitrates, mercury oxycyanide and free mineral acids; for the separation of iron and manganese; the estimation of strychnine and zinc; and the differentiation of the naphthols.

Potassium ferrocyanide in solution is sensitive to light by which it is gradually decomposed. Standardized and test solutions should therefore be kept in dark amber-colored bottles.

Maximum Limits of Impurities

Carbonates (CO_3)	0.0000%
Sulphates (SO_4)	0.0100%
Chlorides (Cl)	0.0200%

Methods of Testing

Carbonates.—Treat the powdered potassium ferrocyanide with diluted sulphuric acid. No gas should be evolved.

Sulphates.—Dissolve 1 gm. in 20 mils of water, add 1 mil of hydrochloric acid and some barium chloride solution. No change should be apparent.

Chlorides.—Deflagrate a mixture of 0.5 gm. of the powdered potassium ferrocyanide with 1 gm. of potassium nitrate by introducing small quantities at a time into a porcelain crucible heated to redness. Treat the residue with 20 mils of water, filter, to the filtrate add 0.5 gm. of potassium nitrate and evaporate to dryness in a porcelain crucible. Fuse the residue until quiescent, allow to cool, dissolve the melt in 20 mils of water and add 3 mils of nitric acid and some silver nitrate solution. The liquid should not develop more than an opalescence.

The potassium nitrate used in this test should comply with the requirements as to chlorides, chlorates and perchlorates under Potassium Nitrate, page 274.

Quantitative Method.—Dissolve about 1 gm., accurately weighed, in 200 mils of water, add 10 mils of sulphuric acid and titrate with tenth-normal potassium permanganate solution until the color changes from yellowish-green to yellowish-red. At least 99% of $\text{K}_4\text{Fe}(\text{CN})_6 + 3\text{H}_2\text{O}$ should be found.

1 mil of tenth-normal $\text{KMnO}_4 = 0.042238$ gm. of
 $\text{K}_4\text{Fe}(\text{CN})_6 + 3\text{H}_2\text{O}$, log 62570.

POTASSIUM HYDROXIDE**KOH****Mol. Wt. 56.11**

Potassium hydroxide is usually in the form of white, very hygroscopic sticks or pieces, having a crystalline fracture. It is soluble in about 1 part of water and readily soluble in alcohol; the small amount of potassium carbonate present may remain undissolved in the alcohol.

The hydroxide and its solutions when exposed to the air readily absorb carbon dioxide and the reagent always contains small amounts of potassium carbonate. Its solutions gradually corrode their glass containers and acquire a content of silicate, aluminum and other constituents of the glass, due note of which must sometimes be taken when using the solutions in tests for these substances.

On heating, potassium hydroxide melts and at a red heat vaporizes. Nickel or silver crucibles are best adapted to the use of the reagent as a flux; porcelain and platinum are attacked by it.

The reagent is used principally as a neutralizing and alkalizing agent; in the determination of nitrates and sulphur; in gas analysis and organic analysis and synthesis; in preparing volumetric solutions; and saponifying fats and other esters.

Potassium hydroxide should be stored in well-stoppered bottles and exposed to the air as little as possible. It is very caustic, attacks the skin and many other forms of organic matter, such as wood, cork, fabrics, etc., contact with which should be avoided. Paraffin-coated corks are well adapted to stoppering the containers of the solid hydroxide; rubber stoppers are generally satisfactory for its solutions.

Three grades of potassium hydroxide commonly used and adapted to various purposes are here described.

I**POTASSIUM HYDROXIDE, PUREST**

Potassium hydroxide, purest, is usually in the form of irregular pieces. It contains at least 82% of KOH and not more than 2.75% of K_2CO_3 .

Maximum Limits of Impurities

Sulphates (SO_4)	0.0040%
Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0016%
Nitrites (N_2O_3)	0.0001%
Nitrogen as Nitrates, Nitrites, Ammonia, etc. (N) ..	0.0014%
Phosphates (P_2O_5)	0.0040%
Silicates (SiO_2)	0.0200%
Aluminum (Al)	0.0106%
Calcium (Ca)	0.0120%
Heavy Metals	0.0000%
Potassium Carbonate (K_2CO_3)	2.7500%

Methods of Testing

Sulphates.—Dissolve 3 gm. in 50 mls of water, acidulate the solution with 7 to 8 mls of hydrochloric acid, heat to boiling, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 20 mls of water, acidulate with 5 mls of nitric acid and add silver nitrate solution. Not more than a slight opalescence should result.

Nitrates.—Dissolve 2 gm. in a mixture of 8 mls of water and 2 mls of sulphuric acid, add a few milligrams of sodium chloride and 1 drop of indigo solution and mix the solution with 10 mls of sulphuric acid. The blue color should persist after mixing.

Nitrites.—Dissolve 1 gm. in 10 mls of water and add 10 mls of 10% sulphuric acid and some zinc iodide-starch solution. No blue color should be produced.

Nitrogen as Nitrates, Nitrites, Ammonia, etc.—Dissolve 50 gm. in 200 mls of water in a distilling flask, add a mixture of 1 gm. each of zinc dust and powdered iron by hydrogen (or 2 gm. of Devarda's Metal) and connect the flask with a bulb-tube, condenser and a receiver containing 10 mls of tenth-normal hydrochloric acid solution. Allow the mixture to stand one to two hours, distill off about 50 mls and titrate the distillate with tenth-normal sodium hydroxide solution, using methyl orange as indicator. Not

more than 0.5 mil of the tenth-normal acid solution should be neutralized by the ammonia in the distillate.

If an apparent excess of nitrogen be found, the reagents should be tested for nitrogen and the amount found deducted from the total indicated by the previous test. Or another test may be made as described, using the same quantities of reagents but double the amount of the potassium hydroxide under examination. The difference between the amounts of acid neutralized in the two determinations will indicate the amount of nitrogen in 50 gm. of the potassium hydroxide.

Phosphates.—Dissolve 5 gm. in 50 mils of water, add 25 mils of nitric acid and 25 mils of ammonium molybdate solution and allow to stand two hours at about 40° C. No yellow precipitate should form.

Aluminum.—Dissolve 5 gm. in 10 mils of water, add 25 mils of 36% acetic acid and then ammonia water in slight excess, dilute with water to 100 mils and heat on a steam-bath until the odor of ammonia has disappeared. Add 2 or 3 drops more of ammonia water and allow it to stand twelve to eighteen hours. Collect any precipitate which may have formed upon an ashless filter (save the filtrate for further tests) wash, ignite and weigh it. The weight should not exceed 0.001 gm.

Calcium and Heavy Metals.—Divide the filtrate obtained in the preceding test into two equal parts. To one add ammonium oxalate solution and allow it to stand two to three hours. No precipitate should form. To the other portion of the filtrate add a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Silicates.—Dissolve 5 gm. in 25 mils of water in a platinum dish, add 25 mils of hydrochloric acid, evaporate the solution to dryness on a steam-bath and dry the residue one-half hour at about 120° C. Dissolve the residue in 100 mils of water, acidulated with 10 mils of hydrochloric acid and collect, wash, ignite and weigh any insoluble residue which may be left. The weight should not exceed 0.001 gm.

Quantitative Method and Determination of Potassium Carbonate.—Dissolve the potassium hydroxide in water and titrate the solution, without heat or excessive shaking, with standard hydrochloric acid solution, using phenolphthalein as indicator. When the phenolphthalein end-point has been reached note the amount of acid solution used, add methyl orange and titrate further to the end-point for that indicator. At least 82% of KOH and not more than 2.75% of K_2CO_3 should be found.

1 mil of normal HCl = 0.05611 gm. of KOH, log 74904.

1 mil of normal HCl = 0.0691 gm. of K_2CO_3 , log 83948.

Note: The difference between the number of mils of standard acid solution used in the first and second titrations represents the amount of acid required to neutralize the KOH. The number of mils used in the second titration when multiplied by 2, represents the amount of the acid required to neutralize the K_2CO_3 .

II

POTASSIUM HYDROXIDE, PURE

Potassium hydroxide, pure, is usually in the form of sticks and contains at least 80% of KOH and not more than 4% of K_2CO_3 . This grade is often designated as "purified by alcohol."

Maximum Limits of Impurities

Sulphates (SO_4)	0.0040%
Chlorides (Cl)	0.0250%
Nitrates (N_2O_5)	0.0016%
Nitrogen as Nitrates, Nitrites, Ammonia, etc. (N). ..	0.0014%
Silicates (SiO_2)	0.0600%
Aluminum (Al)	0.0320%
Calcium (Ca)	0.0500%
Heavy Metals	Trace
Potassium Carbonate (K_2CO_3)	4.0000%

Methods of Testing

Sulphates, Nitrates and Nitrogen as Nitrates, Nitrites, Ammonia, etc.—Perform the tests as directed

under Potassium Hydroxide, Purest. The results should be as there stated.

Chlorides.—To 20 mls of a 1% solution of the potassium hydroxide add 2 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Silicates.—Perform the test as directed under Potassium Hydroxide, Purest. The weight of the residue obtained should not exceed 0.003 gm.

Aluminum.—Perform the test as directed under Potassium Hydroxide, Purest. The weight of the residue obtained should not exceed 0.003 gm.

Calcium and Heavy Metals.—To the ammoniacal filtrate obtained in the preceding test add ammonium oxalate solution and a few drops of ammonium sulphide solution. No precipitate should form and not more than a slight green color should develop.

Quantitative Method and Determination of Potassium Carbonate.—Make the determinations as directed under Potassium Hydroxide, Purest. At least 80% of KOH and not more than 4% of K_2CO_3 should be found.

III

POTASSIUM HYDROXIDE, PURIFIED

Potassium hydroxide, purified, is usually in the form of white sticks, frequently prepared by electrolysis. It contains at least 80% of KOH and not more than 5% of K_2CO_3 . Chlorides are often present in this grade of the reagent. When potassium hydroxide is required containing a minimum or limited quantity of chloride, Potassium Hydroxide, Purest, or Pure should be used.

Maximum Limits of Impurities

Nitrates (N_2O_5)	0.0016%
Aluminum (Al)	0.1000%
Calcium (Ca)	0.1000%
Heavy Metals	Trace
Potassium Carbonate (K_2CO_3)	5.0000%

Methods of Testing

Nitrates.—Perform the test as directed under Potassium Hydroxide, Purest. The result should be as there stated.

Aluminum.—Dissolve 2.5 gm. in 50 mls of water, add 15 mls of 36% acetic acid and then make the solution slightly alkaline with ammonia water. No precipitate should form.

Calcium and Heavy Metals.—To the ammoniacal solution obtained in the preceding test add ammonium oxalate solution and a few drops of ammonium sulphide solution. The former should cause no turbidity at once and the latter should produce at most a slight green color.

Quantitative Method and Determination of Potassium Carbonate.—Make the determinations as described under Potassium Hydroxide, Purest. At least 80% of KOH and not more than 5% of K_2CO_3 should be found.

POTASSIUM HYDROXIDE. SOLUTION, 30%

Solution of potassium hydroxide, 30%, is a clear, colorless liquid having a specific gravity of about 1.3. It contains at least 30% of KOH and not more than 2.0% K_2CO_3 .

The solution is a convenient form for the employment of potassium hydroxide as a precipitant, neutralizer, etc. It should be stored in well-closed, rubber-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_3)	0.0016%
Chlorides (Cl)	0.0200%
Nitrates (N_2O_5)	0.0006%
Nitrogen as Nitrates, Nitrites, Ammonia, etc.	0.0006%
Silicates (SiO_2)	0.0231%
Aluminum (Al)	0.0123%
Calcium (Ca)	0.0200%
Heavy Metals	Trace
Potassium Carbonate (K_2CO_3)	2.000%

Methods of Testing

Sulphates, Nitrates and Nitrogen as Nitrates, Nitrites, Ammonia, etc.—Perform the tests as directed under Potassium Hydroxide, Purest, using 2 mls of the solution for each gm. of the potassium hydroxide directed to be taken. The results should be as there stated.

Silicates and Aluminum.—Perform the tests as directed under Potassium Hydroxide, Purest, using 10 mls of the solution instead of 5 gm. of the solid potassium hydroxide. The weight of the final residue should not exceed 0.003 gm. in each case.

Chlorides.—Dilute 0.4 mil of the solution to 20 mls and add 2 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Calcium and Heavy Metals.—To the ammoniacal filtrate obtained in the test for aluminum add ammonium oxalate solution and a few drops of ammonium sulphide solution. No precipitate should form and not more than a slight green color should develop.

Quantitative Method and Determination of Potassium Carbonate.—Dilute a portion of the solution, accurately weighed, to a convenient volume and perform the titration as described under Potassium Hydroxide, Purest. At least 30% of KOH and not more than 2.0% of K_2CO_3 should be found.

POTASSIUM IODATE

KIO₃

Mol. Wt. 214.02

Potassium iodate is usually in the form of a white, crystalline powder, soluble in about 13 parts of water forming solutions neutral to litmus. It contains at least 99.9% of KIO₃. Heated above 100° C. the salt begins to decompose with liberation of iodine.

Potassium iodate is used principally for preparing volumetric solutions; for determining the value of zinc dust; as a reagent for morphine; and for the determination of zinc and arsenic.

Maximum Limits of Impurities

Free Acids (as HIO_3)	0.0700%
Iodides (I)	0.0015%

Methods of Testing

Free Acids.—Dissolve 0.5 gm. in 20 mls of water which has been thoroughly boiled, add a small crystal of neutral potassium iodide and a few drops of starch solution and allow to stand half a minute. No blue color should develop.

Iodides.—Dissolve 1 gm. in 20 mls of water, add 3 to 5 drops of 10% sulphuric acid and a few mls of chloroform and shake the mixture. The chloroform should not acquire a violet color.

Quantitative Method.—Dry the salt to constant weight over sulphuric acid, weigh out 0.10 to 0.15 gm. of the dried material, dissolve it in 20 mls of water, add 3 gm. of potassium iodide and 5 mls of hydrochloric acid and titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 99.9% of KIO_3 should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.003567$ gm. of KIO_3 ,
log 55230.

POTASSIUM IODIDE**KI**

Mol. Wt. 166.02

Potassium iodide is usually in the form of white, cubical crystals, soluble in less than 1 part of water; also soluble in about 20 parts of alcohol. It is slightly deliquescent in moist air. The salt contains at least 99% of KI and not more than 0.5% of KCl.

Exposed to the air for some time pure potassium iodide is likely to become yellow from liberation of iodine. Traces of potassium carbonate usually present in the article retard this decomposition although an excessive amount of the alkali is undesirable. When the reagent is required to be free from alkali, Potassium Iodide, Neutral, is to be used. Aqueous solutions of the iodide also soon develop free iodine

if the water is not free from oxygen and carbon dioxide. Thoroughly boiled water should therefore be used in making solutions of the reagent. A trace of alkali (0.01%) added to the solution prevents its decomposition for a time.

Potassium iodide is mainly used in the iodometric determination of chlorine, bromine, ferric salts, chromic acid and chromates, arsenic, antimony, peroxides, perborates, persulphates and many other oxidizing substances; in the detection of alkaloids, aldehydes, glucose, albumin, ammonia, caustic alkalis and carbonates, etc.; and in preparing volumetric solutions.

The salt should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Potassium Carbonate (K_2CO_3)	0.0800%
Heavy Metals	0.0000%
Iron (Fe)	0.0010%
Sulphates (SO_4)	0.0100%
Cyanides (CN)	0.0030%
Iodates (I_2O_5)	0.0010%
Nitrates (N_2O_5)	0.0016%
Potassium Chloride (KCl)	0.5000%

Methods of Testing

Potassium Carbonate.—Dissolve 0.5 gm. of potassium iodide in 10 mls of water and add a drop of phenolphthalein solution. No red color should develop.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No color should be produced. Add further a few mls of ammonia water. No brown or green color should develop nor should a precipitate form.

Iron.—Dissolve 2 gm. in 20 mls of water, add a few drops of dilute hydrochloric acid and divide the solution into two equal portions. To one add some potassium sulphocyanate solution. The liquid should not be reddened. To the other portion add a drop of freshly prepared solution of potassium ferricyanide. A bluish or greenish color should not develop.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add a few drops of hydrochloric acid and some barium chloride solution. No change should be apparent.

Cyanides.—Dissolve 10 gm. in 20 mls of water, add a granule of ferrous sulphate, a drop of ferric chloride solution and 5 mls of 27% solution of sodium hydroxide; heat the mixture to about 60° C. and add hydrochloric acid to slight acidulation. No blue color should develop.

Iodates.—Dissolve 1 gm. in 20 mls of water which has been recently boiled and cooled, add a little starch solution and 3 or 4 drops of 10% sulphuric acid and allow the mixture to stand one minute. A blue color should not develop.

Nitrates.—Heat 1 gm. with 5 mls of 27% solution of sodium hydroxide and about 0.2 gm. of aluminum wire and test the vapors with moist red litmus paper. The paper should not become blue.

Potassium Chloride.—In the quantitative determination directed in the following paragraph not more than 60.6 mls of tenth-normal silver nitrate solution should be consumed by each gram of the potassium iodide.

Quantitative Method.—Dissolve about 0.5 gm. of the potassium iodide, accurately weighed, in 25 mls of water, add 20 mls (an excess) of tenth-normal silver nitrate solution, 2 or 3 mls of 10% solution of ferric ammonium sulphate and 2 or 3 mls of nitric acid and titrate the excess of silver nitrate with tenth-normal ammonium sulphocyanate solution. At least 99% of KI should be found.

1 ml of tenth-normal $\text{AgNO}_3 = 0.016602$ gm. of KI,
log 22016.

POTASSIUM IODIDE, NEUTRAL

KI

Mol. Wt. 166.02

Potassium iodide, neutral, is usually in the form of white granules, readily soluble in water and in alcohol. It contains at least 99% of KI and not more than 0.5% of KCl.

This salt on account of its greater freedom from alkali is more sensitive to the action of air and moisture than Potassium Iodide.

Neutral potassium iodide may be used for any of the purposes detailed under Potassium Iodide, and for others requiring unusual freedom from alkalinity. The salt should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Free Alkali (as KOH)	0.0030%
Heavy Metals	0.0000%
Iron (Fe)	0.0010%
Sulphates (SO_4)	0.0100%
Cyanides (CN)	0.0030%
Iodates (I_2O_5)	0.0010%
Nitrates (N_2O_5)	0.0016%
Potassium Chloride (KCl)	0.5000%

Methods of Testing

Free Alkali.—Dissolve 10 gm. of the potassium iodide in 50 mils of well-boiled water in a flask of colorless glass, add 3 drops of iodeosin solution, 1 drop of tenth-normal hydrochloric acid solution and about 30 mils of ether, stopper the flask and shake vigorously. The aqueous liquid should not retain a reddish color.

In applying the test for free alkali the water used should be neutral to iodeosin and should be as free from oxygen as possible. The ether used may at times contain oxidizing substances (peroxides, etc.) which quickly liberate iodine from the solution, making it difficult to discern the point of neutrality to the indicator; such ether is not well adapted to the performance of this test.

Heavy Metals, Iron, Sulphates, Cyanides, Iodates, Nitrates and Potassium Chloride.—The tests for these impurities are to be made as under Potassium Iodide, and the results should be as there stated.

Quantitative Method.—This is to be carried out as described under Potassium Iodide. At least 99% of KI should be found.

POTASSIUM NITRATE**KNO₃****Mol. Wt. 101.11**

Potassium nitrate is usually in the form of colorless crystals, soluble in about 4 parts of water, but almost insoluble in alcohol. Its solutions are neutral to litmus. In moist air the salt is slightly hygroscopic. On ignition the salt is decomposed, oxygen and nitrogen being evolved and oxides of potassium remaining.

Owing to the readiness with which oxygen is yielded on heating, potassium nitrate is much used as a constituent of oxidizing fluxes, especially in determining sulphur in organic substances and silicon in ferro-silicon. It is used in the detection of chromium and manganese, and for other purposes involving oxidation or the destruction of organic matter.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO ₃)	0.0050%
Chlorides (Cl)	0.0010%
Chlorates and Perchlorates (as Cl)	0.0025%
Calcium (Ca)	0.0100%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%
Nitrites (N ₂ O ₃)	0.0001%
Iodates (I ₂ O ₅)	0.0020%

Methods of Testing

Sulphates.—Dissolve 2 gm. in 50 mls of water, add a few drops of hydrochloric acid and some barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with a few drops of nitric acid and add silver nitrate solution. No change should be apparent.

Chlorates and Perchlorates.—Gently ignite 1 gm. of potassium nitrate, dissolve the residue in 20 mls of water and add a few drops of nitric acid and silver nitrate solution. Not more than a very faint opalescence should be apparent.

Calcium.—Dissolve 3 gm. in 50 mls of water and add a few mls of ammonia water and ammonium oxalate solution. No change should be apparent.

Iron.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with a few drops of hydrochloric acid and add potassium sulphocyanate solution. No red color should develop.

Other Heavy Metals.—Dissolve 1 gm. in 10 mls of water and add a few drops each of ammonia water and ammonium sulphide solution. No precipitate or dark color should result.

Nitrates and Iodates.—Dissolve 0.5 gm. in 10 mls of water, add 3 or 4 drops of diluted sulphuric acid and some zinc iodide-starch solution. No blue color should develop within one minute.

POTASSIUM NITRITE

KNO_2

Mol. Wt. 85.11

Potassium nitrite is usually in the form of white or slightly yellowish, hygroscopic, cylindrical sticks. It is very easily soluble in water, forming a solution alkaline to litmus. It contains at least 85% of KNO_2 .

Solutions of potassium nitrite are not stable, gradual oxidation occurring on exposure to the air. The salt sometimes contains traces of carbonate. When it is necessary to remove this impurity from its solutions, this may be done, provided the presence of nitrate is unobjectionable, by adding calcium nitrate solution and filtering off the calcium carbonate formed.

Potassium nitrite is used chiefly in the estimation of cobalt, amino-acids and urea and in the detection of phenols, salicylic acid, iodine, indol, thiophene, phloridzin, mercury cyanide, formaldehyde and antipyrine.

The salt should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Heavy Metals	0.0000%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0100%

Methods of Testing

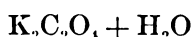
Heavy Metals.—Dissolve 1 gm. of potassium nitrite and 1 gm. of ammonium chloride in 5 mls of water, evaporate to dryness, dissolve the residue in 10 mls of water and add hydrogen sulphide water. No change should be apparent. Add further a slight excess of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with a few drops of hydrochloric acid and add barium chloride solution. No turbidity or opalescence should be produced.

Quantitative Method.—Dissolve about 1 gm., accurately weighed, in water and dilute to 100 mls. Allow 20 mls of this solution to run slowly and with constant shaking into a mixture of 50 mls of tenth-normal potassium permanganate solution, 200 mls of water and 50 mls of 10% sulphuric acid. After ten minutes add 3 gm. of potassium iodide to the mixture and titrate the liberated iodine with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 85% of KNO₂ should be found.

1 mil of tenth-normal KMnO₄ = 0.0042555 gm. of KNO₂,
log 62895.

POTASSIUM OXALATE, NEUTRAL

Mol. Wt. 184.23

Potassium oxalate, neutral, usually occurs as colorless crystals, efflorescent in warm, dry air and soluble in about

3 parts of water. When heated, the salt loses its water of crystallization at about 160° C. At a higher temperature it is decomposed, giving off carbon monoxide and dioxide and leaving a residue of potassium carbonate. The anhydrous oxalate is hygroscopic in moist air.

The reagent is employed chiefly as a precipitant and reducing agent; in testing quinine; and in photography. Its greater solubility than the corresponding ammonium or sodium salts renders it especially suitable for purposes requiring the use of concentrated solutions of a neutral oxalate.

The salt should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Free Acid (as $H_2C_2O_4$)	0.0045%
Free Alkali (as NaOH)	0.0040%
Sulphates (SO_4)	0.0200%
Chlorides (Cl)	0.0050%
Heavy Metals	0.0000%

Methods of Testing

Free Acid and Free Alkali.—Dissolve 10 gm. in 100 mils of water and add phenolphthalein solution. If the solution remains colorless not more than 0.1 mil of tenth-normal sodium hydroxide solution should be required to produce a red color. If reddened by the phenolphthalein, not more than 0.1 mil of tenth-normal hydrochloric acid solution should be required to discharge the color.

Sulphates.—Dissolve 0.5 gm. in 20 mils of water and add 1 mil of hydrochloric acid and some barium chloride solution. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 25 mils of water and add 10 mils of nitric acid and 2 or 3 drops of silver nitrate solution. Not more than a slight opalescence should be produced.

Heavy Metals.—Dissolve 1 gm. in 25 mils of water and add hydrogen sulphide water. No change should be apparent. Then add a few mils of ammonia water and 2 or

3 drops of ammonium sulphide solution. No dark color or precipitate should be produced.

POTASSIUM PERCHLORATE

KClO₄

Mol. Wt. 138.56

Potassium perchlorate usually occurs as colorless crystals, soluble in about 65 parts of water but insoluble in alcohol. It contains at least 99% of KClO₄.

Potassium perchlorate may be distinguished from the chlorate by its failure to yield chlorine when brought in contact with hydrochloric acid; it is not colored by sulphuric acid. It is a strong oxidizing agent, but is not so active as the chlorate. It is, however, subject to decomposition, sometimes violent, by the same agencies that affect the chlorate, viz., heat, concussion, organic matter, oxidizable substances, etc.

The reagent is used chiefly as an oxidizing agent in much the same way as the chlorate. It should be stored in well-stoppered bottles in a cool place, and, like the chlorate, should be handled with caution.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Heavy Metals	0.0000%
Nitrates (N ₂ O ₅)	0.0016%
Sulphates (SO ₄)	0.0100%
Calcium (Ca)	0.0200%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of hot water and add silver nitrate solution. Not more than a slight opalescence should result.

Heavy Metals.—Dissolve 1 gm. in 20 mls of hot water and add hydrogen sulphide water. No change should be apparent. Add a few drops of ammonia water and of ammonium sulphide solution. No dark color or precipitate should be produced.

Nitrates.—Heat 1 gm. with 5 mls of 27% sodium hydroxide solution and 0.5 gm. each of zinc dust and reduced iron (or 1 gm. of Devarda's metal) and test the vapors for ammonia by means of moistened red litmus paper. No ammonia should be evolved.

Sulphates.—Dissolve 1 gm. in 20 mls of hot water and add barium chloride solution. No change should be apparent.

Calcium.—Dissolve 1 gm. in 20 mls of hot water and add a few drops of ammonia water and some ammonium oxalate solution. The liquid should not appear changed.

Quantitative Method.—In a nickel crucible fuse a mixture of about 0.3 gm., accurately weighed, of the potassium perchlorate and 2 gm. of sodium nitrite, free from chlorides, and maintain a state of fusion for about half an hour at a low temperature. Cool, dissolve the melt in about 100 mls of water, add 30 mls (an excess) of tenth-normal silver nitrate solution and 25 mls of nitric acid and warm the mixture on the steam-bath until the red vapors have disappeared. Then add 5 mls of saturated solution of ferric ammonium sulphate and titrate with tenth-normal solution of ammonium sulphocyanate. At least 99% of KClO_4 should be found.

1 mil of tenth-normal $\text{AgNO}_3 = 0.013856$ gm. of KClO_4 ,
log 14163.

POTASSIUM PERMANGANATE

KMnO_4

Mol. Wt. 158.03

Potassium permanganate occurs as dark violet, almost black crystals, exhibiting a steel-blue luster. It is soluble in about 16 parts of water. The reagent contains at least 99% of KMnO_4 .

Potassium permanganate is an efficient oxidizing agent by reason of the ease with which it yields its oxygen. Heated to about 240° it decomposes into oxygen, potassium

manganate and manganese dioxide. Trituration or percussion also effects decomposition. The salt is very sensitive to the action of organic matter by which it is easily reduced, sometimes with explosion. Traces of organic matter in water in which the salt is dissolved soon cause freshly prepared solutions to deteriorate, although when decanted from the sediment which collects on standing a week or two, the clear solution keeps well for a long time if preserved in clean bottles in a dark place.

Potassium permanganate is chiefly used as a volumetric solution in oxidimetry; for the determination of iron, copper, molybdenum, cadmium, mercury, uranium, ferrocyanides, peroxides, sulphides, dextrose, and nitric, nitrous, oxalic and boric acids; as a reagent for eosin, tartaric acid and alkaloids; in testing ammonia water for empyreumatic substances, distinguishing vinegar made by fermentation, differentiating tartaric and citric acids, detecting methyl alcohol in ethyl alcohol, testing benzoin for cinnamic acid, detecting fats in petrolatum and cinnamyl cocaine in cocaine or its salts.

The salt should be stored in well-stoppered, amber-colored bottles in a cool place.

Maximum Limits of Impurities

Sulphates (SO_3)	0.0300%
Chlorides and Chlorates (Cl)	0.0100%
Nitrates	0.0800%

Methods of Testing

Sulphates.—Boil 0.5 gm. with a mixture of 2 or 3 mls of alcohol and 25 mls of water until the reduction is complete, filter the liquid and to the filtrate add 2 mls of nitric acid and some barium nitrate solution. Not more than a slight opalescence should result.

Chlorides and Chlorates.—Heat 0.5 gm. in a crucible, add gradually small pieces of paraffin until the reduction is complete, allow it to cool, treat the residue with 20 mls

of water and filter. To the filtrate add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Nitrates.—Dissolve 0.5 gm. in 5 mls of water at 50° to 60° C., add gradually to the solution 1 gm. of crystallized oxalic acid, heat to boiling and filter. Mix 2 mls of the colorless filtrate with 2 mls of sulphuric acid and overlay the mixture with 1 mil of ferrous sulphate solution. No dark color should develop at the plane of contact of the liquids.

Quantitative Method.—Dry the salt to constant weight over sulphuric acid, weigh accurately about 0.15 gm. of the dried material, dissolve it in 25 mls of water, add 1 mil of sulphuric acid and 50 mls of tenth-normal solution of oxalic acid, warm the solution to about 80° C. and titrate it with tenth-normal solution of potassium permanganate. At least 99% of KMnO_4 should be found.

1 mil of tenth-normal $\text{H}_2\text{C}_2\text{O}_4 = 0.0031606$ gm. of KMnO_4 ,
log 49977.

POTASSIUM PERMANGANATE, FREE FROM SULPHATES

KMnO_4

Mol. Wt. 158.03

Potassium permanganate, free from sulphates, has the appearance and general properties given under Potassium Permanganate. It contains at least 99% of KMnO_4 .

This reagent is suitable for any of the purposes mentioned under Potassium Permanganate as well as others requiring greater freedom from sulphates. It should be stored in well-stoppered, amber-colored bottles in a cool place.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0030%
Chlorides and Chlorates (Cl)	0.0100%
Nitrates (N_2O_5)	0.0800%

Methods of Testing

Sulphates.—Dissolve 3 gm. in 150 mls of water, add 15 mls of alcohol and heat until the liquid is completely decolorized. Filter, add to the filtrate 2 mls of hydrochloric acid and some barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Chlorides, Chlorates and Nitrates.—Perform the tests as directed under Potassium Permanganate. The results should be as there stated.

Quantitative Method.—Make the quantitative determination as directed under Potassium Permanganate. At least 99% of KMnO_4 should be found.

1 mil of tenth-normal $\text{H}_2\text{C}_2\text{O}_4 = 0.0031606$ gm. of KMnO_4 ,
log 49977.

POTASSIUM PERSULPHATE

$\text{K}_2\text{S}_2\text{O}_8$

Mol. Wt. 270.32

Potassium persulphate usually occurs as colorless crystals, soluble in about 50 parts of water, yielding solutions neutral to litmus. It contains at least 95% of $\text{K}_2\text{S}_2\text{O}_8$. Upon heating the salt, sulphur trioxide and oxygen are evolved. Its solutions slowly decompose yielding oxygen and forming an acid sulphate. Heat hastens this change. Exposed to moist air the salt gradually undergoes the same decomposition.

Potassium persulphate is a useful oxidizing agent for removal of organic matter in analysis; in the separation of chlorine, bromine and iodine; in oxidase investigations; and in organic analysis and synthesis.

The reagent should be kept dry by storage in well-stoppered bottles in a cool place and should be exposed to the air as little as possible.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Heavy Metals	Trace

Methods of Testing

Chlorides.—Dissolve 1 gm. in 50 mls of water and add 3 to 5 drops of silver nitrate solution. Not more than a slight opalescence should result. Avoid an excess of silver nitrate which would cause a black precipitate to form.

Heavy Metals.—Dissolve 2 gm. in 25 mls of sulphurous acid with the aid of heat, evaporate to dryness and dissolve the residue in 40 mls of water. To 20 mls of the solution add hydrogen sulphide water. No change should be apparent.

To the remainder of the solution add a slight excess of ammonia water and a few drops of ammonium sulphide solution. A green color may be produced but no precipitate should form at once.

Sulphurous acid may at times contain traces of iron which would react in this test. When the acid, free from iron, is not available, substitute for it a solution of 1 gm. of sodium bisulphite in 25 mls of water.

Quantitative Method.—Introduce about 0.3 gm., accurately weighed, into a solution of 2 gm. of potassium iodide in 50 mls of water, add 10 mls of 10% sulphuric acid, allow to stand half an hour in a stoppered flask with occasional shaking and titrate the liberated iodine with tenth-normal sodium thiosulphate solution. At least 95% of $K_2S_2O_8$ should be found.

1 mil of tenth-normal $Na_2S_2O_3 = 0.013516$ gm. of $K_2S_2O_8$,
log 13085.

POTASSIUM PHOSPHATE, MONOBASIC, ANHYDROUS

(Soerensen's Potassium Phosphate)

KH_2PO_4

Mol. Wt. 136.16

Potassium phosphate, monobasic, anhydrous is usually in the form of colorless crystals, soluble in about 4 parts of water. Its solutions have an acid reaction to litmus. The salt may contain at most 0.1% of moisture.

In applying the tests directed under the methods of testing the water used should always be as nearly free from carbon dioxide as possible. It is advisable to prepare it specially for the purpose not long before the tests are to be made. This may be done by redistilling the ordinary distilled water, rejecting the first portion of the distillate and collecting the remainder in a receiver from which the carbon dioxide has been removed by a current of air, free from carbon dioxide. The redistilled water is protected from contamination by means of soda-lime tubes until it is used.

This reagent is intended especially for use in connection with Sodium Phosphate, Dibasic, Anhydrous, in determining the hydrogen ion concentration of bacteriological culture media and other fluids. Regarding the subject see Clark and Lubs, *The Colorimetric Determination of Hydrogen Ion Concentration and its Applications in Bacteriology*, Jour. of Bacteriology, 2, 1, 109, 191 (1917).

The salt should be stored in tightly-stoppered bottles.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Sulphates (SO ₃)	0.0050%
Chlorides (Cl)	0.0020%
Moisture	0.1000%

Methods of Testing*

Insoluble Matter.—Dissolve 5 gm. in 100 mils of water. No insoluble residue should be left.

Sulphates.—Dissolve 5 gm. in 100 mils of water, add 5 mils each of hydrochloric acid and barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 5 gm. of the salt in 100 mils of water, add 5 mils of nitric acid, sp. gr. 1.40–1.42, and some silver nitrate solution. Not more than a slight opalescence should be produced.

* These methods of testing are adapted from Soerensen's original article, *Ergebnisse der Physiologie*, 12, 433 (1912).

Moisture.—Dry about 2 gm., accurately weighed, in a dessicator over sulphuric acid until the weight is constant. The loss in weight should not exceed 0.1%.

Loss on Ignition (Proof of Proper Composition).—Dry the salt to constant weight over sulphuric acid, weigh out accurately about 2 gm. of the dried material and ignite it carefully to constant weight. The loss on ignition should be not less than 13.13% nor more than 13.33%.

Test for Proper Hydrogen Ion Concentration.—For this test the following special reagents are required:

Citric Acid. The reagent described under Acid Citric on page 14 is tested further as follows: (a) Determine the water content by drying to constant weight at not above 90° C. The loss should be not less than 8.48% and not more than 8.68%. (b) Titrate about 0.4 gm., accurately weighed, with fifth-normal barium hydroxide solution, using phenolphthalein as indicator. From 99.66% to 100.33% of $\text{H}_2\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$ should be found.

Sodium Citrate Solution. Dissolve 21.008 gm. of the citric acid described in the preceding paragraph in 200 mls of normal solution of sodium hydroxide, free from carbonate, and dilute with water to 1 liter.

Solution of the Sodium Salt of Parabenzenesulphonic Acid Azoalphanaphthylamine. A solution containing 0.1 gm. of the chemical in 1 liter of 60% alcohol.

The test is performed as follows: Dissolve 0.9 gm. of the potassium phosphate, monobasic, anhydrous, in water and dilute to 100 mls. Place in test-tubes 3 portions of 10 mls each of this solution and for comparison use 5 other tubes containing the following mixtures:

No. 1—6 mls of sodium citrate solution + 4 mls of tenth-normal hydrochloric acid solution.

No. 2—7 mls of sodium citrate solution + 3 mls of tenth-normal hydrochloric acid solution.

No. 3—7.5 mls of sodium citrate solution + 2.5 mls of tenth-normal hydrochloric acid solution.

No. 4—8 mls of sodium citrate solution + 2 mls of tenth-normal hydrochloric acid solution.

No. 5—9 mls of sodium citrate solution + 1 mil of tenth-normal hydrochloric acid solution.

To each of the 8 tubes add 10 drops of the solution of the sodium salt of parabenzenesulphonic acid-azoalphanaphthylamine and compare the colors of the liquids. The color of the tubes of potassium phosphate solution should lie between those of No. 2 and No. 4 of the citrate mixtures. The addition of 1 drop of tenth-normal hydrochloric acid solution to one of the tubes of phosphate solution and of 1 drop of tenth-normal sodium hydroxide solution to another should shift the colors sharply beyond the boundaries established by control tubes Nos. 2 and 4.

POTASSIUM SULPHATE

K_2SO_4

Mol. Wt. 174.26

Potassium sulphate is usually in the form of white, hard crystals. It is soluble in about 10 parts of water but insoluble in alcohol. Its aqueous solution is neutral to litmus.

Under ordinary conditions the salt is not hygroscopic, but exposed for some time to moist air it slowly deliquesces. When heated the crystals decrepitate and on ignition at moderate temperatures do not undergo change, but at high temperatures the salt is volatilized.

Potassium sulphate is chiefly used as a precipitant for lead and barium and in the Kjeldahl-Gunning-Arnold nitrogen determination as an addition to the digestion mixture.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0010%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%
Calcium (Ca)	0.0200%
Magnesium (Mg)	0.0010%
Nitrates (N_2O_5)	0.0032%
Ammonium Salts (NH_3)	0.0050%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add silver nitrate solution. No change should be apparent.

Iron.—Dissolve 1 gm. in 20 mls of water, add a few drops of nitric acid, boil the solution and add potassium sulphocyanate solution. No red color should develop.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent.

Calcium and Magnesium.—Dissolve 2 gm. in 40 mls of water, add a few mls of ammonia water, to half the solution add ammonium oxalate solution and to the other half ammonium phosphate solution. No change should be apparent in either case.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add a few milligrams of sodium chloride, 1 drop of indigo solution and 10 mls of sulphuric acid and shake the mixture. The blue color should persist after shaking.

Ammonium Salts.—Dissolve 1 gm. in 20 mls of water and add 0.5 ml of Nessler's solution. At most a slight yellow color should result.

POTASSIUM SULPHOCYANATE**(Potassium Thiocyanate)**

KCNS

Mol. Wt. 97.18

Potassium sulphocyanate is usually obtained in the form of colorless, deliquescent crystals, easily soluble in water (less than 1 part) and in alcohol. The complete solubility of the salt in alcohol serves to detect other less soluble salts such as chlorides, sulphates, etc.

Potassium sulphocyanate is used principally in the detection of iron, copper, silver and molybdic acid; in the determination of halogens, copper and mercury; in quantitative and qualitative reagents for glucose; and in the detection of albumin and free mineral acids.

The reagent should be kept in tightly-stoppered containers and should not be exposed to contamination with even traces of iron, such as might occur in dust. Sometimes iron existing in the glass of bottles containing the salt will discolor it after long storage.

Maximum Limits of Impurities

Substances Insoluble in Alcohol	0.0000%
Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0200%
Heavy Metals	0.0000%
Iron (Fe)	0.0005%
Ammonium Salts (NH ₄)	0.0025%

Methods of Testing

Substances Insoluble in Alcohol.—Dissolve 1 gm. of potassium sulphocyanate in 10 mils of boiling alcohol. The solution should be clear and free from undissolved residue.

Chlorides.—Dissolve 1 gm. in 100 mils of water, add 20 mils of nitric acid, heat on the steam-bath for 3 hours and then add silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 0.5 gm. in 20 mils of water and add 1 to 2 mils of hydrochloric acid and some barium chloride solution. No change should be apparent.

Heavy Metals.—Dissolve 1 gm. in 20 mils of water and add a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Iron.—Dissolve 1 gm. in 20 mils of water and add a few drops of hydrochloric acid. The solution should remain colorless.

Ammonium Salts.—Heat to boiling a mixture of 2.5 gm. of potassium sulphocyanate and 10 mils of 27% sodium hydroxide solution. The vapors should not change the color of moistened red litmus paper.

POTASSIUM TETRAOXALATE



Potassium tetraoxalate is usually in the form of colorless crystals, soluble in about 55 parts of cold water and readily in boiling water. The aqueous solution is acid to litmus. The salt contains at least 99.5% of $\text{KHC}_2\text{O}_4 + \text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$.

The salt is used chiefly as a starting material for the preparation of volumetric solutions for acidimetry and oxidimetry. It is well-adapted to this purpose as its composition can be determined by titration by standard alkali and by permanganate, thus eliminating error which might arise from the presence of an excess of the binoxalate, small amounts of which are often present in the commercial article. Although it effloresces when heated, the tetraoxalate is stable at ordinary temperatures and can therefore be used in the air-dried condition. Sodium oxalate and sodium carbonate as starting materials for permanganate and acid solutions respectively, are generally preferred, but potassium tetraoxalate is a very useful substance for the purpose once its reducing and neutralizing value have been accurately established.

The reagent should be stored in well-stoppered bottles and not exposed to extremes of heat.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Sulphates (SO_4)	0.0050%
Heavy Metals	0.0000%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 25 mls of water with the aid of 10 mls of nitric acid and mix the solution with 2 or 3 drops of silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 5 gm. in 250 mls of water, add 10 mls of hydrochloric acid, heat to boiling, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Heavy Metals.—Dissolve 1 gm. in 25 mls of warm water and add hydrogen sulphide water. No change should be apparent. After adding ammonia water until the reaction is alkaline add a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Quantitative Method.—The determination is to be made by both acidimetric and oxidimetric means. At least 99.5% of $\text{KHC}_2\text{O}_4 + \text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$ should be found in each case.

Dissolve about 0.3 gm., accurately weighed, in 50 mls of water and titrate the solution, while boiling, with tenth-normal sodium hydroxide solution, using phenolphthalein as indicator.

1 mil of tenth-normal $\text{NaOH} = 0.0084727$ gm. of
 $\text{KHC}_2\text{O}_4 + \text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$, log 92803

Dissolve about 0.2 gm., accurately weighed, in 25 mls of water, add 5 or 6 mls of sulphuric acid, warm the mixture to about 60° C. and titrate with tenth-normal potassium permanganate solution.

1 mil of tenth-normal $\text{KMnO}_4 = 0.0063545$ gm. of
 $\text{KHC}_2\text{O}_4 + \text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$, log 80309.

POTASSIUM AND SODIUM TARTRATE

(Rochelle Salt; Seignette Salt)

$\text{KNaC}_4\text{H}_4\text{O}_6 + 4\text{H}_2\text{O}$ Mol. Wt. 282.22

Potassium and sodium tartrate is usually in the form of clear, colorless crystals, readily soluble in water but almost insoluble in alcohol. It is slightly efflorescent in dry air. Its aqueous solution is alkaline to litmus but is neutral to phenolphthalein.

The salt is used mainly in the preparation of alkaline copper solutions employed in the detection and determination of reducing sugars. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Calcium (Ca)	0.0300%
Heavy Metals	0.0000%
Chlorides (Cl)	0.0010%
Sulphates (SO ₄)	0.0625%
Ammonium Compounds (NH ₃)	0.0045%

Methods of Testing

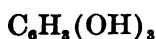
Calcium.—Dissolve 1 gm. in 10 mls of water, add 5 mls of 36% acetic acid and allow crystals to deposit, shaking the mixture if necessary to induce crystallization. Filter, dilute the filtrate with an equal volume of water, add 8 to 10 drops of ammonium oxalate solution and allow to stand one minute. No turbidity should develop.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent. Add a few drops of ammonium sulphide solution. No green or brown color should develop nor should a precipitate form.

Chlorides.—Dissolve 1 gm. in 10 mls of water and add 5 mls of nitric acid and some silver nitrate solution. No change should be apparent.

Sulphates.—Dissolve 2 gm. in 45 mls of water and add 5 mls of hydrochloric acid and some barium chloride solution. No turbidity should be produced.

Ammonium Compounds.—Dissolve 1 gm. in 10 mls of water, add 10 mls of 27% sodium hydroxide solution and heat the mixture to boiling. The vapors should not turn red litmus paper blue.

PYROGALLOL**(Pyrogalllic Acid)**

Mol. Wt. 126.08

Pyrogallol usually occurs as light, white, lustrous crystals, readily soluble in about 1.7 parts of water, about 1 part of

alcohol or about 1.5 parts of ether, but with difficulty in chloroform, benzene or carbon disulphide. It melts at about 131° C. and its aqueous solution is neutral or only slightly acid to litmus.

On exposure to air and light the substance becomes discolored, acquiring a grayish tint. Its solutions quickly deteriorate, becoming brown and developing an acid reaction; this color change is due to the absorption of oxygen and is greatly accelerated by the presence of alkalis.

Pyrogallol is an active reducing agent, reducing solutions of the salts of gold, silver and mercury even in the cold. The reagent is used chiefly in gas analysis, an alkaline solution being employed for absorbing oxygen; as a reagent for copper, gold, hydrated chloral, sulphonmethane, lignin and sesame oil and for carbon monoxide in blood; in determining nitric acid and nitrous acids; and in detecting propeptone and cell membrane.

Pyrogallol should be stored in well-stoppered bottles of amber glass. Its solutions should be freshly prepared when required.

Maximum Limits of Impurities

Inorganic Matter	0.0500%
Gallic Acid	1.0000%

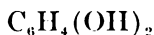
Methods of Testing

Inorganic Matter.—Ignite 2 gm. of the pyrogallol. The weight of the ash should not exceed 0.001 gm.

Gallic Acid.—Dissolve 2 gm. in 5 mls of ether of specific gravity 0.720. No insoluble matter should remain.

RESORCINOL

(Resorcin)



Mol. Wt. 110.08

Resorcinol usually occurs as colorless, needle-shaped crystals, having a slight peculiar odor. It is soluble in about 1 part of water or alcohol, easily soluble in ether, but difficultly in chloroform. The aqueous solution is neutral

or only faintly acid to litmus. Resorcinol melts at 109° to 111° C. (theory 110° C.) and, when heated higher, sublimes. Exposed to air and light it acquires a pink color; the presence of ammonia vapor hastens the discoloration.

The reagent is used principally in the detection of chloral, chloroform, hydrochloric acid in gastric juice, sugars, saccharin, asparagin, naphthalene, aldehydes, iodoform, sesame and cottonseed oils, tartaric and chloric acids, nitrates, nitrites, zinc and copper salts and of cotton in woolen textiles; also in the examination of certain phenols and in the determination of cineol.

Resorcinol should be stored in well-stoppered bottles and protected from light.

Maximum Limits of Impurities

Non-volatile Matter	0.0500%
Diresorcin	0.0000%
Phenol	0.0000%
Free acids	0.0000%

Methods of Testing

Non-volatile Matter.—Ignite 2 gm. of resorcinol and weigh the residue. The weight should not exceed 0.001 gm.

Diresorcin and Phenol.—Dissolve 1 gm. in 20 mls of water. The solution should be perfectly clear. Then warm the liquid and note the odor. It should not have a phenolic odor.

Free Acids.—Dissolve 1 gm. in 10 mls of alcohol and add a drop of lacmoid solution. The liquid should assume a blue color, or if the color is reddish, 1 drop of tenth-normal solution of sodium hydroxide should be sufficient to produce a distinct blue color.

SACCHAROSE

(Sucrose)



Mol. Wt. 342.24

Saccharose usually occurs as small, colorless crystals or white granules, soluble in about 0.5 part of water and in

about 200 parts of alcohol; insoluble in chloroform or ether. Its solutions are neutral to litmus and are dextrorotary. The specific rotation of saccharose should be not less than $+66.4^{\circ}$ at 20° C.

Saccharose is easily inverted by heating with dilute acids or alkalis or even to some extent by heat alone. Therefore when a weight of dry saccharose is required, as in polariscopy and other exacting work, it is advisable to determine the moisture in a separate portion and introduce a correction for the amount found.

Saccharose is used principally for standardizing solutions used in determining reducing sugars; and in the detection of alkaloids, fats, albumins, thymol, bile acids and sesame oil.

Maximum Limits of Impurities

Insoluble Substances	0.0000%
Non-volatile Substances	0.0100%
Sulphates (SO_4)	0.0050%
Chlorides (Cl)	0.0010%
Heavy Metals	0.0000%
Invert Sugar	0.1000%

Methods of Testing

Insoluble Substances.—Dissolve 10 gm. in 50 mls of water. The solution should be clear and free from undissolved matter.

Non-volatile Substances.—Ignite 5 gm. and weigh the ash. The weight should not exceed 0.0005 gm.

Sulphates.—Dissolve 2 gm. in 20 mls of water and add barium chloride solution. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add silver nitrate solution. No change should be apparent.

Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent. Then add a few drops each of ammonia water and ammonium sulphide solution. No dark color or precipitate should be produced.

Invert Sugar.—Dissolve 10 gm. in 50 mls of water in a 250-mil beaker, add 50 mls of Fehling's solution, heat the mixture at such a rate that about four minutes are required to bring it to a boil, and then boil for exactly two minutes. Add at once 100 mls of water which has been recently boiled and then cooled, collect any cuprous oxide which may have formed by filtration through a weighed Gooch crucible with an asbestos pad, wash well with water, followed successively by 10 mls each of alcohol and ether, dry the crucible at 100° C. and weigh. The weight of cuprous oxide should not exceed 0.070 gm.

In order to determine the percentage of invert sugar from the weight of the copper oxide, consult Hertzfeld's table, U. S. Dept. of Agriculture, Bureau of Chemistry, Bulletin No. 107, Revised, 43 (1912).

Determination of Specific Rotation.—Determine the moisture in the saccharose by drying at 105° C. to constant weight. Dissolve an accurately weighed amount of the original saccharose, corresponding to 26 gm. of anhydrous saccharose, in water, dilute to a volume of 100 mls at 20° C. and determine the specific rotation $(\alpha)_D$ at the same temperature. A value for $(\alpha)_D$ of at least 66.4° should be found.

SEA SAND

Sea sand is in the form of fine granules which have been washed with acid and subsequently ignited. The reagent is used principally as a diluent of substances undergoing extraction, ignition, drying or other operation.

Maximum Limits of Impurities

Substances Soluble in Hydrochloric Acid	0.1200%
Chlorides (Cl)	0.0050%
Volatile Substances	0.0200%

Methods of Testing

Substances Soluble in Hydrochloric Acid.—Digest 20 gm. with a mixture of 20 mls of hydrochloric acid and

100 mls of water with frequent shaking for about four hours on the steam-bath, replacing the loss by evaporation. Filter, evaporate 50 mil of the filtrate, ignite the residue and weigh it. The weight should not exceed 0.01 gm.

Chlorides.—Shake 1 gm. with 20 mls of water, filter and add silver nitrate solution to the filtrate. Not more than a slight opalescence should result.

Volatile Substances.—Ignite 5 gm. of sea sand and determine the loss in weight. The loss should not exceed 0.001 gm.

SILVER

Ag

At. Wt. 107.88

Silver is a white, lustrous metal, usually occurring in the form of sheets, sometimes as granules or powder. It is soluble in nitric and hot sulphuric acids, insoluble in hydrochloric or cold, dilute sulphuric acid. It contains at least 99.97% of Ag.

Silver is employed principally as a standard for preparing volumetric solutions; in elementary analysis; and in the determination of halogens. The metal should be stored in well-closed bottles and should not be exposed to the fumes of hydrogen sulphide or acids.

Maximum Limit of Impurity

Foreign Metals 0.0250%

Foreign Metals.—Dissolve 2 gm. in 10 mls of nitric acid. The solution should be clear and free from undissolved residue. Dilute the solution with 50 mls of water. No turbidity should be produced. Heat the solution to boiling, add a slight excess of hydrochloric acid and boil until the precipitate becomes flocculent and settles out on standing, leaving the liquid clear. Filter, evaporate the filtrate to dryness and weigh the residue. The weight should not exceed 0.0005 gm.

Quantitative Method.—Dissolve about 0.2 gm., accurately weighed, in 5 mls of nitric acid and evaporate the

solution to dryness. Dissolve the residue in a little water, add 2 gm. of sodium cyanide, transfer to a weighed platinum dish, dilute to about 40 mls and electrolyze the solution, using the platinum dish as the cathode, rotating the anode about 500 revolutions per minute and passing a current of about 1.5 amperes and 5 to 7 volts until the silver is completely precipitated. Finally interrupt the current, remove the electrolyte, wash the deposit of silver with water, dry it at 100° C. and weigh. At least 99.97% of Ag should be found.

SILVER NITRATE

AgNO₃

Mol. Wt. 169.89

Silver nitrate is usually in the form of colorless crystals, soluble in less than 1 part of water; also soluble in alcohol. It contains at least 99.9% of AgNO₃.

Silver nitrate is not easily affected by light when in pure condition, but in the presence of organic matter, even in traces (dust) it quickly darkens on exposure to light. It is readily reduced, especially in alkaline solution, by a large number of substances, such as aldehydes, sugars, tartaric and formic acids, etc.

Silver nitrate is used mainly in detecting and determining halogens, cyanides, phosphates, arsenates and sulphocyanates; for detecting aldehydes, reducing sugars, cottonseed and mustard oils, chromates, uric acid, arsine, carbon monoxide, hydrogen peroxide, artificial camphor and a variety of other substances; in the assay of mustard oil; and in the examination of glycerin and other materials.

The reagent should be protected from contamination with organic matter by storage in glass-stoppered bottles of amber glass. Its standardized solutions should be stored away from the light.

Maximum Limits of Impurities

Chlorides (Cl)	0.0020%
Substances Insoluble in Alcohol	0.0000%
Copper (Cu)	0.0050%
Bismuth (Bi)	0.0600%
Lead (Pb)	0.0600%
Substances not Precipitated by Hydrochloric Acid.	0.0500%

Methods of Testing

Chlorides.—Dissolve 5 gm. of silver nitrate in 5 mls of water and pour the solution into 100 mls of water, acidulated with 2 mls of nitric acid. No turbidity or opalescence should appear.

Substances Insoluble in Alcohol (Potassium Nitrate, Etc.).—Dissolve 0.5 gm. in 0.5 ml of water, mix the solution with 20 mls of absolute alcohol and agitate the mixture. No turbidity should result.

Copper, Bismuth and Lead.—Dissolve 5 gm. in 20 mls of water and add ammonia water until the precipitate first formed is redissolved. The liquid should be clear and colorless.

Substances not Precipitated by Hydrochloric Acid.—Dissolve 2 gm. in 20 mls of water, heat the solution to boiling and add 3 mls of hydrochloric acid. Allow the precipitate to settle, filter, evaporate the filtrate to dryness and weigh the residue. The weight should not exceed 0.001 gm.

Quantitative Method.—Dissolve about 0.5 gm., accurately weighed, in 10 mls of water, add a solution of 2 gm. of sodium cyanide in 10 mls of water, transfer the solution to a platinum dish, dilute to about 50 mls and electrolyze the solution, using a current of about 2.5 amperes and about 5 to 7 volts and rotating the anode about 500 revolutions per minute. When the silver is completely deposited upon the platinum dish cathode (usually about twenty minutes are required) shut off the current, remove the electrolyte, wash the deposited silver with water and then with alcohol, dry at about 100° C. and weigh. The amount of silver found deposited on the cathode should be equivalent to at least 99.9% of AgNO_3 .

$$\text{Ag} \times 1.57481 = \text{AgNO}_3, \log 19703.$$

SODIUM

Na

At. Wt. 23.0

Metallic sodium is at ordinary temperature a soft, waxy solid. Its freshly cut surfaces are silver white but on exposure to air quickly become dull gray and finally accumulate a crust consisting of oxide, hydroxide and carbonate. It melts at about 95° C. (theory 97.6° C.).

Brought in contact with water a violent reaction takes place, sodium hydroxide being formed with liberation of hydrogen. When heated strongly in the air it takes fire and burns to the oxide.

Metallic sodium is used mainly as a reducing agent, both alone and in the form of sodium amalgam; for the detection of nitrogen, sulphur, phosphorus and halogens in organic compounds; for the detection of water, alcohol, nitric acid, sulphurous acid, and arsenic; for differentiating alpha and beta naphthols and detecting nitrobenzene in bitter almond oil; as a flux; and in preparing volumetric solutions.

The reagent should be stored in well-closed, glass-topped bottles containing sufficient liquid petrolatum or other dry, neutral, petroleum oil to cover the metal completely. Sodium should not be exposed to the air for any length of time and should not be handled with the bare fingers or with moist instruments. Contact with water should be carefully avoided.

Maximum Limits of Impurities

Nitrogen (N)	0.0700%
Heavy Metals	0.0000%

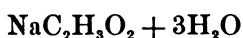
Methods of Testing

Nitrogen.—Free 1 gm. of sodium from any adhering oil by means of filter paper, cut the metal into small pieces and throw them one at a time upon 20 mls of cold water. Warm the solution so obtained. No odor of ammonia should be perceptible.

Heavy Metals.—To the solution obtained in the preceding test add a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Dissolve 1 gm. in 20 mls of water as directed under the test for nitrogen, slightly acidulate the solution with hydrochloric acid and add hydrogen sulphide water. No change should be apparent.

SODIUM ACETATE



Mol. Wt. 136.08

Sodium acetate is usually in the form of colorless, transparent crystals, efflorescent in warm air. It is easily soluble in water and moderately in alcohol. The 5% aqueous solution of the salt is neutral or slightly alkaline to litmus or phenolphthalein.

Sodium acetate is chiefly used for the detection of narcotine, papaverine and narceine and the quantitative separation of opium alkaloids; for precipitating iron and aluminum; and in the determination of phosphoric acid.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0010%
Sulphates (SO ₃)	0.0100%
Calcium (Ca)	0.0200%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 1 mil of nitric acid and some silver nitrate solution. No change should be apparent.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add a few drops of hydrochloric acid and some barium chloride solution. No turbidity should result.

Iron.—Dissolve 1 gm. in 20 mls of water add 1 mil of hydrochloric acid and some potassium sulphocyanate solution. The mixture should not have a red color.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent.

Calcium.—Dissolve 1 gm. in 20 mls of water and add ammonium oxalate solution. No change should be apparent.

SODIUM AMALGAM

$\text{Na} + \text{Hg}$

Sodium amalgam usually occurs as soft, gray masses or granules, containing at least 1.5% and usually about 2% of Na. The consistence of the amalgam depends upon the percentage of sodium present. An amalgam containing 1% of sodium is a thick fluid; one with 1.25%, a pasty mass; while one containing 1.5% or more is solid.

The reagent is employed principally as a reducing agent in organic synthesis; for detecting sulphur dioxide, strychnine, sulphonmethane and water or alcohol in ether; and for preparing hydrogen.

Sodium amalgam should be stored in tightly-closed, glass-stoppered bottles. It should be kept from contact with water or moist air by which it is quickly decomposed.

Method of Testing

Quantitative Method.—Introduce about 10 gm., accurately weighed, into 100 mls of water, allow it to stand with repeated shaking until the evolution of hydrogen has entirely ceased and titrate with normal hydrochloric acid, using methyl orange as indicator. At least 1.5% of Na should be found.

1 mil of normal $\text{HCl} = 0.0230$ gm. of Na, log 36173.

SODIUM BICARBONATE

NaHCO_3 ,

Mol. Wt. 84.01

Sodium bicarbonate usually occurs as a white, crystalline powder, soluble in about 12 parts of water but insoluble in

alcohol. The reagent contains at least 99.9% of NaHCO_3 .

A freshly prepared solution of sodium bicarbonate in cold water is slightly alkaline to litmus. The alkalinity increases on standing, shaking or heating, due to the loss of carbon dioxide and formation of sodium carbonate. The dry salt when heated to a little below 300°C . changes to the normal carbonate and this process is employed in obtaining anhydrous sodium carbonate suitable for use as a starting material for preparing volumetric solutions. The temperature should not exceed 300°C . however, as the carbonate begins to dissociate at somewhat above that temperature.

Sodium bicarbonate is used chiefly for the preparation of volumetric solutions; in the detection of alkaloids; and as a general neutralizing agent.

The salt should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0025%
Silicates	0.0000%
Chlorides (Cl)	0.0050%
Thiosulphates ($\text{Na}_2\text{S}_2\text{O}_3$)	0.0065%
Phosphates (P_2O_5)	0.0150%
Iron (Fe)	0.0015%
Other Heavy Metals	0.0000%
Potassium	Trace
Monocarbonate	Trace
Ammonium Compounds (NH_3)	0.0032%
Sulphocyanates (CNS)	0.0200%

Methods of Testing

Sulphates.—Dissolve 2 gm. in 30 mls of water, slightly acidify the solution with hydrochloric acid, heat to boiling, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Silicates.—Dissolve 5 gm. of sodium bicarbonate in a mixture of 15 mls of water and 20 mls of hydrochloric acid and evaporate the solution to dryness in a platinum dish. Dry the residue at 120°C . for half an hour and then dissolve it in 25 mls of water acidulated with 2 to 3 mls

of hydrochloric acid. The solution should be perfectly clear.

Chlorides and Thiosulphates.—Dissolve 1 gm. in 40 mls of water and add 3 mls of nitric acid. The solution should be clear. Then add silver nitrate solution and allow it to stand ten minutes. Not more than a slight opalescence should result.

Phosphates.—Dissolve 5 gm. in 50 mls of water, add 10 mls of nitric acid and 25 mls of ammonium molybdate solution and allow it to stand two hours at about 40° C. No yellow precipitate should form.

Iron.—Dissolve 1 gm. in 15 mls of water, slightly acidulate with nitric acid, boil a moment and add potassium sulphocyanate solution. Not more than a slight red color should result.

Other Heavy Metals.—Dissolve 3 gm. in 40 mls of water, slightly acidulate with hydrochloric acid and add hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No brown or green color should develop and no precipitate should form.

Potassium.—Hold a particle of the salt in a non-luminous flame and view the flame through cobalt glass. Not more than a transient violet tinge should be observed.

Monocarbonate (Normal Sodium Carbonate).—Dissolve 1 gm. in 20 mls of water at a temperature below 15° C. and without excessive shaking, and add 3 drops of phenolphthalein solution. Not more than a faint red color should be produced.

Ammonium Compounds.—Heat 1 gm. in a test-tube and test the vapor with moistened red litmus paper. The paper should not turn blue.

Sulphocyanates.—Dissolve 5 gm. in 50 mls of water acidulated with 10 mls of nitric acid and add 1 drop of ferric chloride solution. No red color should be produced.

Quantitative Method.—Dry the salt over sulphuric acid to constant weight, weigh out a sample of the dried material, dissolve it in water and titrate the solution with a standard acid solution, using methyl orange as indicator. At least 99.9% of NaHCO_3 should be found.

1 mil of normal HCl = 0.08401 gm. of NaHCO_3 , log 92433.

SODIUM BISMUTHATE

Sodium bismuthate is usually obtained as a yellow to brown, amorphous, somewhat hygroscopic powder. It is not a true bismuthate but has a variable composition. It is insoluble in water, slowly soluble in nitric acid, quickly when heated. Sodium bismuthate is somewhat unstable. It is decomposed by boiling water, quite rapidly if acidulated. The reagent contains at least 4% of active O, corresponding to at least 70% of NaBiO_3 .

The reagent is used principally for the determination of manganese in iron, steel and ores. Regarding its employment for this purpose see Treadwell-Hall's *Analytical Chemistry*, 4th ed., II, 616 (1915), or Scott's *Standard Methods of Chemical Analysis*, 2d ed., revised, 263 (1918).

Sodium bismuthate should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0025%
Manganese (Mn)	0.0010%

Methods of Testing

Chlorides.—Heat to boiling a mixture of 1 gm. of sodium bismuthate and 10 mls of water and filter. To the perfectly clear filtrate add a few drops of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Manganese.—Dissolve 1 gm. in 10 mls of nitric acid with the aid of a moderate heat. The solution should be almost colorless and should not have a red or violet tint.

Quantitative Method.—Weigh accurately about 3 gm. of sodium bismuthate, mix it in a 100-mil graduated flask with a solution of 20 gm. of potassium iodide in 30 mils of water and shake well. Then add 20 mils of hydrochloric acid, stopper the flask well, allow it to stand one hour in the dark, dilute to the mark, mix and titrate an aliquot portion of the solution with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 4% of active O should be found in the sodium bismuthate.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.0008$ gm. of active O.

SODIUM BISULPHATE

(Sodium Acid Sulphate)



Mol. Wt. 138.08

Sodium bisulphate usually occurs as colorless crystals, hygroscopic in moist air. It is easily soluble in water (less than 2 parts) forming solutions acid to litmus. The reagent contains at least 95% of $\text{NaHSO}_4 + \text{H}_2\text{O}$. On ignition the salt loses water and sulphuric acid and leaves behind a residue of sodium sulphate.

The reagent is used chiefly as a flux in the decomposition of minerals. It should be stored in well-stoppered bottles. Glass stoppers are preferable as the salt slowly attacks and destroys such materials as cork, paper, etc.

Maximum Limits of Impurities

Heavy Metals	0.0000%
Chlorides (Cl)	0.0050%
Arsenic (As)	0.0015%
Potassium	Trace

Methods of Testing

Heavy Metals.—Dissolve 1 gm. in 20 mils of water and add hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Chlorides.—Dissolve 1 gm. in 20 mils of water and add silver nitrate solution. Not more than a slight opalescence should be produced.

Arsenic.—Mix 1 gm. with 3 mils of stannous chloride solution and allow it to stand one hour. The mixture should not acquire a dark color.

Potassium.—Observe through cobalt glass the flame produced by the salt. Not more than a transient violet color should be apparent.

Quantitative Method.—Dissolve the salt in water and titrate the solution with standard alkali solution, using methyl orange as indicator. At least 95% of $\text{NaHSO}_4 + \text{H}_2\text{O}$ should be found.

1 mil of normal $\text{NaOH} = 0.13808$ gm. of $\text{NaHSO}_4 + \text{H}_2\text{O}$,
log 14013.

SODIUM BISULPHITE

(Sodium Acid Sulphite)

NaHSO_3

Mol. Wt. 104.07

Sodium bisulphite usually occurs as a white, granular powder, having the odor of sulphur dioxide. It is soluble in about 4 parts of water and in about 80 parts of alcohol, yielding solutions acid to litmus. It contains at least 90% of NaHSO_3 . The salt, when exposed to the air, loses sulphur dioxide and gradually oxidizes to the sulphate. Its solutions also undergo the same change.

Sodium bisulphite is used chiefly as a reducing agent, in the determination of aldehydes and ketones and in the assay of oils containing cinnamic aldehyde or citral.

The salt should be stored in a cool place in well-stoppered bottles containing as little air space as possible.

Maximum Limits of Impurities

Chlorides	0.0100%
Heavy Metals	Trace
Arsenic	0.0005%

scribed leaves 52.9%. The loss in each case is the water of crystallization.

The reagent is used principally in blowpipe analysis and as a flux in metallurgical work; in volumetric analysis; and as a reagent for curcuma, aloes, glycerin and polyatomic alcohols.

Sodium borate should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Carbonates	0.0000%
Sulphates (SO ₃)	0.0100%
Chlorides (Cl)	0.0010%
Calcium (Ca)	0.0200%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%

Methods of Testing

Carbonates.—Add hydrochloric acid to a cold saturated solution of sodium borate. No effervescence should result.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add 2 mls of hydrochloric acid and some barium chloride solution. No change should result.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 3 mls of nitric acid and some silver nitrate solution. No change should be apparent.

Calcium.—Dissolve 1 gm. in 20 mls of water and add ammonium oxalate solution. No turbidity should result.

Iron.—Dissolve 1 gm. in 20 mls of water and add 2 mls of hydrochloric acid and some potassium sulphocyanate solution. A red color should not develop.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add 2 mls of hydrochloric acid and some hydrogen sulphide water. No change should be apparent.

Quantitative Method.—Dissolve the sodium borate in water and titrate the solution with a standard acid solution, using methyl orange as indicator. At least 99% of Na₂B₄O₇ + 10H₂O should be found.

1 mil of normal HCl = 0.19108 gm. of Na₂B₄O₇ + 10H₂O,
log 28121.

faintly opalescent and no crystalline precipitate should form.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add 1 mil of hydrochloric acid and some barium chloride solution. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

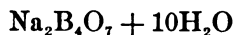
Heavy Metals.—Dissolve 2 gm. in 40 mls of water. To half the solution add hydrogen sulphide water and to the other half a slight excess of ammonia water and a few drops of ammonium sulphide solution. No precipitate or dark coloration should result in either case.

Quantitative Method.—Dissolve about 5 gm., accurately weighed, in 100 mls of water, boil the solution and, while boiling, titrate it with normal sodium hydroxide solution, using phenolphthalein as indicator. Not less than 99% of $\text{NaHC}_4\text{H}_4\text{O}_6 + \text{H}_2\text{O}$ should be found.

1 mil of normal $\text{NaOH} = 0.19008$ gm. of $\text{NaHC}_4\text{H}_4\text{O}_6 + \text{H}_2\text{O}$, log 27893.

SODIUM BORATE, CRYSTALS

(Borax; Sodium Biborate; Sodium Tetraborate)



Mol. Wt. 382.16

Sodium borate crystals are hard, white, prismatic crystals, soluble in about 20 parts of water, yielding a solution alkaline to litmus. It is soluble in about 2 parts of glycerin and in less than 1 part of boiling water but is practically insoluble in alcohol. The salt is efflorescent in dry air. It contains at least 99% of $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$.

Sodium borate containing five molecules of water of crystallization is known and may be distinguished from the salt here described by its octahedral form and by the higher percentage of residue remaining after ignition; it leaves theoretically 69.2% of residue, while the reagent here de-

scribed leaves 52.9%. The loss in each case is the water of crystallization.

The reagent is used principally in blowpipe analysis and as a flux in metallurgical work; in volumetric analysis; and as a reagent for curcuma, aloes, glycerin and polyatomic alcohols.

Sodium borate should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Carbonates	0.0000%
Sulphates (SO ₃)	0.0100%
Chlorides (Cl)	0.0010%
Calcium (Ca)	0.0200%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%

Methods of Testing

Carbonates.—Add hydrochloric acid to a cold saturated solution of sodium borate. No effervescence should result.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add 2 mls of hydrochloric acid and some barium chloride solution. No change should result.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 3 mls of nitric acid and some silver nitrate solution. No change should be apparent.

Calcium.—Dissolve 1 gm. in 20 mls of water and add ammonium oxalate solution. No turbidity should result.

Iron.—Dissolve 1 gm. in 20 mls of water and add 2 mls of hydrochloric acid and some potassium sulphocyanate solution. A red color should not develop.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add 2 mls of hydrochloric acid and some hydrogen sulphide water. No change should be apparent.

Quantitative Method.—Dissolve the sodium borate in water and titrate the solution with a standard acid solution, using methyl orange as indicator. At least 99% of Na₂B₄O₇ + 10H₂O should be found.

1 mil of normal HCl = 0.19108 gm. of Na₂B₄O₇ + 10H₂O,
log 28121.

SODIUM BORATE, CALCINED

Sodium borate, calcined, is usually in the form of white, spongy masses or a white powder. It is soluble in about 30 parts of water but insoluble in alcohol. It contains at least 75% of $\text{Na}_2\text{B}_4\text{O}_7$.

The reagent is crystallized sodium borate from which a large portion of the water of crystallization has been removed by heat. It is on this account, and also by reason of its friable nature, more suitable than the crystals for certain purposes for which a less hydrated salt may be desired, as for instance, in blow-pipe analysis and fusions.

The salt should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Carbonates	0.0000%
Sulphates (SO_4)	0.0150%
Chlorides (Cl)	0.0015%
Calcium (Ca)	0.0285%
Iron (Fe)	0.0012%
Other Heavy Metals	0.0000%

Methods of Testing

Carbonates, Sulphates, Chlorides, Calcium, Iron, Other Heavy Metals.—Perform the tests as directed under Sodium Borate, Crystals, using 0.7 gm. of the calcined sodium borate instead of 1 gm. of the crystals. The results should be as there stated.

Quantitative Method.—Perform the titration as directed under Sodium Borate, Crystals. At least 75% of $\text{Na}_2\text{B}_4\text{O}_7$ should be found.

1 mil of normal HCl = 0.1010 gm. of $\text{Na}_2\text{B}_4\text{O}_7$, log 00432.

SODIUM BROMATE

NaBrO_3

Mol. Wt. 150.92

Sodium bromate occurs usually as colorless crystals, sometimes as a crystalline powder, soluble in about 3 parts of water. It contains at least 99.8% of NaBrO_3 .

When mixed with a bromide in acid solution bromine is quantitatively liberated, hence the salt is chiefly used in this way in reactions involving oxidation or bromination, such as the determination of resorcin, phenol and phosphorous acid and the liberation of iodine from iodides. Sodium bromate is also used as a starting material for volumetric solutions used in oxidimetry.

Maximum Limit of Impurity

Bromides (Br) 0.0300%

Methods of Testing

Bromides.—Dissolve 2 gm. in 20 mls of water and add a few drops of diluted sulphuric acid. A yellow color should not be produced at once.

Quantitative Method.—Dissolve 0.1 to 0.15 gm. in 20 mls of water, add 3 gm. of potassium iodide and 5 mls of hydrochloric acid and titrate the liberated iodine with tenth-normal solution of sodium thiosulphate. At least 99.8% of NaBrO_3 should be found.

1 mil of tenth-normal $\text{Na}_2\text{S}_2\text{O}_3 = 0.0025153$ gm. NaBrO_3 ,
log 40059.

SODIUM CARBONATE, CRYSTALS

$\text{Na}_2\text{CO}_3 + 10\text{H}_2\text{O}$

Mol. Wt. 286.16

Sodium carbonate, crystals, occurs as colorless, transparent crystals, readily efflorescing on exposure to the air. The salt is soluble in about 1.6 parts of water and yields solutions strongly alkaline to litmus. It contains about 37% of anhydrous Na_2CO_3 .

Salts crystallizing with varying amounts of water, from one to fifteen molecules, are known. The content of about 37% of anhydrous sodium carbonate distinguishes the salt of the composition indicated by the chemical formula given above. Sodium carbonate, crystals, melts at about 33°C . When rendered anhydrous by drying, the salt has the properties described under Sodium Carbonate, Anhydrous.

The reagent is used chiefly as a neutralizing and alkalizing agent and as a precipitant for various metals. In general sodium carbonate precipitates solutions of all metals except the alkalis.

The reagent should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Substances Insoluble in Water	0.0000%
Sodium Hydroxide (NaOH)	0.1000%
Sulphates (SO ₄)	0.0015%
Chlorides (Cl)	0.0010%
Silicates	0.0000%
Nitrates (N ₂ O ₅)	0.0016%
Phosphates (P ₂ O ₅)	0.0010%
Potassium	Trace
Ammonium Compounds (NH ₃)	0.0001%
Calcium (Ca)	0.0020%
Magnesium (Mg)	0.0002%
Iron (Fe)	0.0001%
Other Heavy Metals	0.0000%
Arsenic (As)	0.00005%

Methods of Testing

Substances Insoluble in Water.—Dissolve 20 gm. in 80 mls of water. A clear, colorless solution should result.

Sodium Hydroxide.—Only well-boiled water should be used in this test. Dissolve 3 gm. in 50 mls of water, add a solution of 6 gm. of barium chloride dissolved in 30 mls of water and dilute the mixture to exactly 100 mls. Thoroughly shake the mixture, filter and to 50 mls of the filtrate add phenolphthalein solution. A red color should not be produced.

Sulphates.—Dissolve 10 gm. in 50 mls of water, add a slight excess of hydrochloric acid, boil the solution, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Chlorides.—Dissolve 5 gm. in 50 mls of water, add a slight excess of nitric acid and some silver nitrate solution. No change should be apparent.

Silicates.—Dissolve 20 gm. in a mixture of 30 mils of hydrochloric acid and 20 mils of water, and evaporate the solution in a platinum dish on the steam-bath to dryness. Dry the residue at 120° C. for half an hour and then dissolve it in a mixture of 3 mils of hydrochloric acid and 50 mils of water. The solution should be perfectly clear.

Nitrates.—Dissolve 2 gm. in 10 mils of diluted sulphuric acid, add a few milligrams of sodium chloride, a drop of indigo solution and 10 mils of sulphuric acid and shake the mixture. The blue color should persist after shaking.

Phosphates.—Dissolve 20 gm. in 50 mils of nitric acid, add 50 mils of ammonium molybdate solution and allow it to stand two hours at about 40° C. No yellow precipitate should form.

Potassium.—Place some of the salt in a non-luminous flame and view it through a cobalt glass. At most a transient violet tinge should be apparent.

Ammonium Compounds.—Dissolve 10 gm. in 50 mils of water and add 1 mil of Nessler's solution. No yellow or brown color should result.

Calcium and Magnesium.—Dissolve 10 gm. in 10 mils of water, add 10 mil of hydrochloric acid and then ammonia water until the solution is ammoniacal; then add ammonium oxalate and ammonium phosphate solutions and allow it to stand two to three hours. No precipitate should form.

Iron.—Dissolve 10 gm. in 25 mils of water, slightly supersaturate the solution with hydrochloric acid and add potassium sulphocyanate solution. No red color should develop.

Other Heavy Metals.—Dissolve 20 gm. in 50 mils of water, slightly supersaturate the solution with hydrochloric acid and add hydrogen sulphide water. No change should be apparent. Then make the solution alkaline with ammonia water and add a few drops of ammonium sulphide solution. No precipitate or dark color should result.

Arsenic.—Dissolve 30 gm. in a mixture of 20 mls of sulphuric acid and 80 mls of water, introduce the solution in small quantities at a time into a Marsh apparatus and allow the action to proceed for one hour after the solution is all added. No deposit of arsenic should be visible in the reduction tube.

Quantitative Method.—Dissolve 3 to 4 gm. of the sodium carbonate, accurately weighed, in 50 mls of water and titrate the solution with normal solution of hydrochloric acid, using methyl orange as indicator. About 37% of Na_2CO_3 should be found.

1 mil of normal HCl = 0.053 gm. of Na_2CO_3 , log 72428.

SODIUM CARBONATE, ANHYDROUS

Na_2CO_3

Mol. Wt. 106.0

Anhydrous sodium carbonate is a white, hygroscopic powder, soluble in about 6 parts of water. It contains at least 99% of Na_2CO_3 .

When ignited the salt melts, carbon dioxide being lost. At lower temperatures, even at 400°C ., some loss of carbon dioxide occurs, so that a temperature of 300°C . must not be exceeded when drying the salt for use as a volumetric standard.

Anhydrous sodium carbonate is principally used as a flux, both alone and combined with potassium carbonate, nitrate, etc., in the analysis of silicates, titanates, organic substances, and in detecting manganese and chromium; in blow-pipe analysis; as a starting material for volumetric solutions; and for the usual purposes for which Sodium Carbonate, Crystals, is employed. For use as a flux anhydrous sodium carbonate is often combined with potassium carbonate in molecular proportions, the mixture having a lower fusion point than either of its constituents.

The reagent should be stored in well-stoppered bottles and especially protected from contact with moist air from which it quickly abstracts water.

Maximum Limits of Impurities

Substances Insoluble in Water	0.0000%
Sodium Hydroxide (NaOH)	0.3000%
Sulphates (SO_4)	0.0045%
Chlorides (Cl)	0.0030%
Silicates	0.0000%
Nitrates (N_2O_5)	0.0050%
Phosphates (P_2O_5)	0.0030%
Potassium	Trace
Ammonium Compounds (NH_3)	0.0003%
Calcium (Ca)	0.0060%
Magnesium (Mg)	0.0006%
Iron (Fe)	0.0003%
Other Heavy Metals	0.0000%
Arsenic (As)	0.00015%

Methods of Testing

Perform the tests as directed under Sodium Carbonate, Crystals, but in each test use only one-third as much of the anhydrous sodium carbonate as of the crystals. The results should be as indicated in each test under Sodium Carbonate, Crystals.

Quantitative Method.—Dissolve 1 to 2 gm. of the anhydrous sodium carbonate, accurately weighed, in 50 mls of water and titrate the solution with normal solution of hydrochloric acid, using methyl orange as indicator. At least 99% of Na_2CO_3 should be found.

1 mil of normal HCl = 0.0530 gm. of Na_2CO_3 , log 72428.

SODIUM CHLORIDE

NaCl

Mol. Wt. 58.46

Sodium chloride occurs as white crystals or a crystalline powder, slightly hygroscopic in moist air and soluble in about 3 parts of water. Its aqueous solution is neutral to litmus. The salt contains at least 99.5% of NaCl. At a red heat the salt melts and at the same time volatilizes to some extent.

Sodium chloride is chiefly used in the determination of silver by gravimetric and volumetric means; in the detec-

tion of food colors; and in the metallurgy of silver, gold and mercury.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0020%
Iodides (I)	0.0300%
Calcium (Ca)	0.0100%
Magnesium (Mg)	0.0010%
Potassium (K)	0.1800%
Ammonium Salts (NH_4)	0.0005%
Iron (Fe)	0.0005%
Other Heavy Metals	0.0000%

Methods of Testing

Sulphates.—Dissolve 3 gm. in 20 mls of water. The solution should be clear. Dilute to 100 mls, add 1 mil of hydrochloric acid, heat to boiling, add barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Iodides.—Dissolve 5 gm. in 20 mls of water and add a drop of ferric chloride solution and some starch solution. No blue color should result.

Calcium.—Dissolve 3 gm. in 50 mls of water and add a few drops of ammonia water and some ammonium oxalate solution. No change should be apparent.

Magnesium.—Dissolve 3 gm. in 10 mls of water, add 5 mls of ammonia water and some ammonium phosphate solution and allow it to stand two to three hours. No precipitate should form.

Potassium.—Dissolve 1 gm. in 10 mls of water, add 5 mls of sodium-cobaltic nitrite solution and allow it to stand twelve to eighteen hours. No precipitate should form.

Ammonium Salts.—Dissolve 2 gm. in 20 mls of water and add 1 mil of Nessler's solution. No change should be apparent.

Iron.—Dissolve 2 gm. in 20 mls of water, add a few drops of nitric acid, heat to boiling and add potassium sulphocyanate solution. No red color should be produced.

Other Heavy Metals.—Dissolve 1 gm. in 10 mls of water and add successively 10 mls of hydrogen sulphide water and a slight excess of ammonia water. No change should be apparent after each addition.

Quantitative Method.—Dry the salt to constant weight at 100° C., dissolve a weighed portion of the dried salt in water and titrate with standard silver nitrate solution, using potassium chromate as indicator. At least 99.5% of NaCl should be found.

1 mil of tenth-normal $\text{AgNO}_3 = 0.005846$ gm. of NaCl,
log 76686.

SODIUM CHLORIDE, FUSED

NaCl

Mol. Wt. 58.46

Fused sodium chloride usually occurs as white, translucent, irregular pieces or granules. It dissolves in about 3 parts of water and contains at least 99.5% of NaCl.

The reagent is used chiefly for producing sodium light for polariscopic and other work as well as for the usual purposes for which Sodium Chloride is employed. It should be kept in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0020%
Iodides (I)	0.0300%
Calcium (Ca)	0.0100%
Magnesium (Mg)	0.0010%
Potassium (K)	0.1800%
Ammonium Salts (NH_4)	0.0005%
Iron (Fe)	0.0005%
Other Heavy Metals	0.0000%

Methods of Testing

Perform the tests as directed under Sodium Chloride. The results should be as there indicated.

Quantitative Method.—Dry the salt and perform the titration as directed under Sodium Chloride. At least 99.5% of NaCl should be found.

SODIUM CYANIDE

NaCN

Mol. Wt. 49.01

Sodium cyanide usually occurs as white, deliquescent granules or lumps, readily soluble in water and slightly soluble in alcohol, yielding solutions alkaline to litmus. It contains at least 95% of NaCN.

The salt is practically odorless in dry condition but in moist air an odor of hydrocyanic acid is perceptible, due to the gradual liberation of the acid by the carbon dioxide of the air. On long exposure the salt gradually changes to sodium carbonate, especially when warm. Solutions undergo the same change on standing exposed to the air; ammonia and a formate are generated on boiling. Acids readily liberate from the salt or its solutions the very poisonous hydrocyanic acid, a light and volatile gas. Operations involving the acidulation of more than very small quantities of the salt should therefore be carried out under a well-ventilated hood.

The reagent is used principally as a reducing agent in the determination of antimony, tin, arsenic, nickel, cobalt and copper; in separating bismuth from copper and nickel from cobalt; as a reagent for copper, cobalt, thiosulphates, glucose, gallic acid, santonin, aloes and for carbon monoxide in blood; and in electrolysis.

The salt should be stored in well-stoppered bottles and should be protected from light, moist air and acid fumes.

Maximum Limits of Impurities

Sulphides (S)	0.0030%
Carbonates	Small Amount
Sulphocyanates (CNS)	0.0150%
Ferrocyanides (Fe[CN] ₆)	0.0050%
Sulphates (SO ₄)	0.0100%
Chlorides (Cl)	0.0100%

Methods of Testing

Sulphides.—Dissolve 1 gm. in 20 mls of water and add lead acetate solution. A pure white precipitate should be produced.

Carbonates.—Add hydrochloric acid in excess to a 5% solution of the sodium cyanide under a hood. Not more than a slight effervescence should be visible.

Sulphocyanates and Ferrocyanides.—Dissolve 1 gm. in 20 mls of water, add 3 to 4 mls of hydrochloric acid and a drop of ferric chloride solution. No red or blue color should develop.

Sulphates.—Dissolve 1 gm. in 20 mls of water and add 2.5 mls of hydrochloric acid and some barium chloride solution. No turbidity should result.

Chlorides.—Dissolve 0.5 gm. in 20 mls of water and add 30 mls of 5% silver nitrate solution and 10 mls of nitric acid. Filter off the precipitate, wash it with water, dry at 100° C. and ignite in a porcelain crucible without the filter paper. To the residue add 25 mls of 10% sulphuric acid and a few granules of zinc, let it stand two or three hours with frequent shaking, filter and to the filtrate add nitric acid and silver nitrate solution. Not more than a slight opalescence should be produced.

Quantitative Method.—Dissolve about 0.4 gm. accurately weighed, in 25 mls of water, add 4 mls of ammonia water and a few drops of potassium iodide solution and titrate with tenth-normal silver nitrate solution to the production of a permanent precipitate. At least 95% of NaCN should be found.

1 ml of tenth-normal $\text{AgNO}_3 = 0.009802$ gm. of NaCN,
log 99131.

SODIUM HYDROXIDE

NaOH

Mol. Wt. 40.01

Sodium hydroxide occurs usually as white, deliquescent sticks or pieces, exhibiting a crystalline fracture. It is

soluble in less than 1 part of water, also readily soluble in alcohol; the small amount of sodium carbonate present may remain undissolved in the alcohol.

Exposed to the air the hydroxide besides taking up moisture readily absorbs carbon dioxide and is gradually transformed to the carbonate. Small amounts of carbonate are always present in the reagent. When heated, sodium hydroxide melts and at a red heat volatilizes. Nickel or silver crucibles are preferred for igniting it as porcelain and platinum are attacked by it at red heat. The hydroxide, particularly in solution, gradually attacks glass and dissolves from it silica and other constituents which must sometimes be considered in using the reagent.

Sodium hydroxide is used mainly as a precipitant and neutralizer; for absorbing carbon dioxide; in gas analysis; and for preparing volumetric solutions.

The reagent should be stored in well-stoppered bottles. Cork stoppers should be paraffined or otherwise protected; rubber stoppers are preferable for its solutions. The hydroxide and its strong solutions attack most organic matter and are very caustic; contact with organic bodies or the skin should be avoided.

For various purposes three grades of sodium hydroxide are usually employed, differing from each other in degree of purity. Their descriptions follow.

I

SODIUM HYDROXIDE, FROM SODIUM

Sodium hydroxide, from sodium, usually occurs as white, hygroscopic fragments. It contains at least 95% of NaOH and not more than 3% of Na_2CO_3 .

Maximum Limits of Impurities

Sulphates (SO_3)	0.0040%
Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0016%
Nitrites (N_2O_3)	0.0001%
Nitrogen as Nitrates, Nitrites, Ammonia, etc. (N).	0.0014%

Phosphates (P_2O_5)	0.0040%
Silicates (SiO_2)	0.0200%
Aluminum (Al)	0.0011%
Calcium (Ca)	0.0120%
Heavy Metals	0.0000%
Sodium Carbonate (Na_2CO_3)	3.0000%

Methods of Testing

Sulphates.—Dissolve 3 gm. in 50 mils of water, acidulate the solution with 10 mils of hydrochloric acid, heat to boiling, add barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 20 mils of water, acidulate with 10 mils of nitric acid and add silver nitrate solution. Not more than a slight opalescence should result.

Nitrates.—Dissolve 2 gm. in a mixture of 8 mils of water and 2 mils of sulphuric acid, add a few milligrams of sodium chloride and 1 drop of indigo solution and mix the solution with 10 mils of sulphuric acid. The blue color should persist after the mixing.

Nitrites.—Dissolve 1 gm. in 10 mils of water and add 15 mils of 10% sulphuric acid and some zinc iodide-starch solution. No blue color should be produced.

Nitrogen as Nitrates, Nitrites, Ammonia, etc.—Dissolve 50 gm. in 200 mils of water in a distilling flask, add a mixture of 1 gm. each of zinc dust and powdered iron by hydrogen (or 2 gm. of Devarda's metal) and connect the flask with a bulb-tube, condenser and a receiver containing 10 mils of tenth-normal hydrochloric acid solution. Allow the mixture to stand one to two hours, distil off about 50 mils and titrate the distillate with tenth-normal sodium hydroxide solutions, using methyl orange as indicator. Not more than 0.5 mil of the tenth-normal acid solution should be neutralized by the ammonia in the distillate.

If an apparent excess of nitrogen is found, the reagents should be tested for nitrogen and the amount found in them deducted from the total obtained in the previous test. Or

another test may be made using the same quantities of the reagents but double the amount of the sodium hydroxide under examination. The difference between the amounts of acid neutralized in the two determinations indicates the amount of nitrogen in 50 gm. of the sodium hydroxide.

Phosphates.—Dissolve 5 gm. in 50 mls of water, add 25 mls each of nitric acid and ammonium molybdate solution and allow to stand two hours at about 40° C. No yellow precipitate should form.

Aluminum.—Dissolve 50 gm. in 250 mls of water, add 85 mls of 99½% acetic acid and then ammonia water until the reaction is strongly alkaline. Heat the mixture on the steam-bath until the odor of ammonia has disappeared, add 2 or 3 drops more of ammonia water and allow to stand twelve to eighteen hours. Collect upon an ashless filter any precipitate which may have formed (save the filtrate for further tests), wash, ignite and weigh it. The weight should not exceed 0.001 gm.

Calcium and Heavy Metals.—Dilute the filtrate obtained in the preceding test to a volume of 400 mls. To 20 mls of the liquid add ammonium oxalate solution and allow it to stand one to two hours. No precipitate should form.

To another portion of 20 mls of the diluted filtrate add a few drops of ammonium sulphide solution. No precipitate or dark color should be produced.

Silicates.—Dissolve 5 gm. in 25 mls of water in a platinum dish, add 25 mls of hydrochloric acid, evaporate the solution to dryness on a steam-bath and dry the residue for half an hour at 120° C. Dissolve the residue in 100 mls of water acidulated with 10 mls of hydrochloric acid and collect, wash, ignite and weigh any insoluble matter which may be left. The weight should not exceed 0.001 gm.

Quantitative Method and Determination of Sodium Carbonate.—Dissolve the sodium hydroxide in water and titrate the solution, without heat or excessive shaking, with

a standard acid solution, using phenolphthalein as indicator. When the phenolphthalein end-point has been reached, note the amount of acid solution used, add methyl orange and titrate further to the end point for that indicator. At least 95% of NaOH and not more than 3% of Na_2CO_3 should be found.

1 mil of normal HCl = 0.04001 gm. NaOH, log 60217.

1 mil of normal HCl = 0.0530 gm. of Na_2CO_3 , log 72428.

Note: The difference between the number of mls of standard acid solution used in the two titrations is the amount required to neutralize the NaOH. The number of mls of the acid solution used in the second titration, when multiplied by 2, represents the amount of the acid required to neutralize the Na_2CO_3 .

II

SODIUM HYDROXIDE, PURE

Sodium hydroxide, pure, is usually in the form of white, hygroscopic sticks. It contains at least 94% of NaOH and not more than 4% of Na_2CO_3 . This grade is often called "purified by alcohol."

Maximum Limits of Impurities

Sulphates (SO_4)	0.0343%
Chlorides (Cl)	0.0250%
Nitrates (N_2O_5)	0.0016%
Nitrogen as Nitrates, Nitrites, Ammonia, etc. (N) ..	0.0014%
Aluminum (Al)	0.0320%
Calcium (Ca)	0.0050%
Heavy Metals	Trace
Silicates (SiO_2)	0.0600%
Sodium Carbonate (Na_2CO_3)	4.0000%

Methods of Testing

Sulphates.—Dissolve 3 gm. in 50 mls of water, add 10 mls of hydrochloric acid, heat to boiling, add barium chloride solution and allow to stand twelve to eighteen hours. Collect, wash, ignite and weigh any precipitate which may form. The weight should not exceed 0.003 gm.

Chlorides.—To 20 mls of a 1% solution of the sodium hydroxide add 3 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Nitrates and Nitrogen as Nitrates, Nitrites, Ammonia, etc.—Perform the tests as directed under Sodium Hydroxide, from Sodium. The results should be as there stated.

Silicates.—Perform the test as directed under Sodium Hydroxide from Sodium. The weight of the residue obtained should not exceed 0.003 gm.

Aluminum.—Dissolve 5 gm. in 50 mls of water, add 25 mls of 36% acetic acid and then ammonia water in slight excess and heat on the steam-bath until free from the odor of ammonia. Add 2 or 3 drops of ammonia water, allow to stand twelve to eighteen hours, filter off any precipitate that forms (reserve the filtrate) and wash, ignite and weigh it. The weight should not exceed 0.003 gm.

Calcium and Heavy Metals.—To the ammoniacal filtrate obtained in the preceding test add ammonium oxalate solution and a few drops of ammonium sulphide solution. No precipitate and at most a slight green color should occur.

Quantitative Method and Determination of Sodium Carbonate.—Make the determinations as directed under Sodium Hydroxide, from Sodium. At least 94% of NaOH and not more than 4% of Na_2CO_3 should be found.

III

SODIUM HYDROXIDE, PURIFIED

Sodium hydroxide, purified, is usually in the form of white, hygroscopic sticks. It contains at least 90% of NaOH and not more than 5.3% of Na_2CO_3 .

Maximum Limits of Impurities

Nitrates and Nitrites (as N_2O_5)	0.0020%
Aluminum (Al)	0.1600%

Calcium (Ca)	0.0100%
Heavy Metals	Trace
Sodium Carbonate (Na_2CO_3)	5.3000%

Methods of Testing

Nitrates and Nitrites.—Dissolve 2 gm. in a mixture of 8 mls of water and 2 mls of sulphuric acid, add a few milligrams of sodium chloride, a drop of indigo solution and 10 mls of concentrated sulphuric acid and shake the mixture. A blue color should persist after the shaking.

Aluminum and Calcium.—Dissolve 2.5 gm. in 100 mls of water, add 15 mls of 36% acetic acid and then a slight excess of ammonia water. No flocculent precipitate should form. Then add to the ammoniacal liquid some ammonium oxalate solution. No turbidity should appear at once.

Heavy Metals.—Dissolve 2 gm. in 50 mls of water, acidulate the solution slightly with hydrochloric acid and add hydrogen sulphide water. No change should be apparent. Then add a slight excess of ammonia water and a few drops of ammonium sulphide solution. Not more than a slight green color should result.

Quantitative Method and Determination of Sodium Carbonate.—Make the determinations as directed under Sodium Hydroxide, from Sodium. At least 90% of NaOH and not more than 5.3% of Na_2CO_3 should be found.

SODIUM HYDROXIDE SOLUTION, 27%

Free from Nitrogen

Sodium hydroxide solution is a clear colorless liquid having a specific gravity not below 1.3 and containing from 27% to 30% of NaOH. The solution is intended especially for use in detecting and determining nitrogen or ammonia, but may be used as an alkalizing agent or precipitant in other operations. It should be stored in well-closed, rubber-stoppered bottles.

Maximum Limit of Impurity

Nitrogen (N)	0.0003%
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Methods of Testing

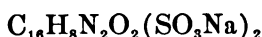
Nitrogen.—Place 200 mls of the sodium hydroxide solution in a distilling flask, add 1 gm. each of iron by hydrogen and zinc dust (or 2 gm. of Devarda's metal), connect the flask with a bulb-tube, a condenser and a receiver containing 10 mls of tenth-normal solution of hydrochloric acid, allow to stand one to two hours, distil off about 75 mls and titrate the distillate with tenth-normal sodium hydroxide solution. Not more than 0.5 mil of the tenth-normal hydrochloric acid solution should be neutralized by the distillate.

Quantitative Method.—Dilute a weighed portion of the solution to a suitable volume and titrate it with a standard acid solution, using methyl orange as indicator. From 27% to 30% of NaOH should be found.

1 mil of normal HCl = 0.04001 gm. of NaOH, log 60217.

SODIUM INDIGOTINSULPHONATE

(Indigo Carmine)



Mol. Wt. 466.28

Sodium indigotinsulphonate is usually in the form of a dark blue powder, soluble in water but almost insoluble in alcohol. It contains, when anhydrous, at least 45% of $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_2$ (indigotin). Sodium indigotinsulphonate sometimes occurs commercially as a paste containing considerable water. The salt deprived of excessive water is the article employed generally for reagent purposes.

The blue color of solutions of the salt is discharged by nitric acid, chlorine or bromine; also by reducing agents such as stannous chloride or a mixture of zinc dust and sodium hydroxide. The salt is used chiefly as a reagent for nitric acid and as a stain in microscopy.

Maximum Limits of Impurities

Water	10.0000%
Insoluble Matter	1.0000%
Starch	0.7500%
Ferrocyanides and Ferricyanides (as CN)	0.0500%

Methods of Testing

Water.—Dry sodium indigotinsulphonate at 100° C. The loss in weight should not exceed 10%.

Insoluble Matter.—Dissolve 1 gm. in 200 mls of water, filter the solution through a tared or counterpoised filter, wash the filter with water until the washings cease to be colored blue, dry the residue at 100° C. and weigh it. The weight should not exceed 0.1 gm.

Starch.—Boil 0.2 gm. of the salt with 10 mls of water, cool the mixture and add bromine water gradually, with agitation, until the blue color has disappeared. Then add a few drops of iodine solution. No blue color should result.

Ferrocyanides and Ferricyanides.—Add 10 mls of 5% sodium hydroxide solution and 15 mls of 3% hydrogen peroxide to 0.25 gm. of the salt, shake the mixture until the blue color disappears, evaporate the liquid to about 10 mls, acidulate with hydrochloric acid and add a few drops of ferrous sulphate solution. No blue color should be produced.

Quantitative Method.—Dry the salt to constant weight at 100° C., dissolve 0.1 to 0.2 gm. of the dried material, accurately weighed, in 10 mls of water, add 1 mil of sulphuric acid, dilute the mixture with 600 mls of water and titrate it in a porcelain dish with tenth-normal potassium permanganate solution. At least 45% of $C_{16}H_{10}N_2O_2$ (indigotin) should be found. The end-point of the titration is the color change from green to light yellow, not the incipient red coloration.

1 mil of tenth-normal $KMnO_4 = 0.007493$ gm. of
 $C_{16}H_{10}N_2O_2$, log 87466.

SODIUM NITRATE

$NaNO_3$

Mol. Wt. 85.01

Sodium nitrate usually occurs as colorless crystals, soluble in a little more than 1 part of water and in about 100

parts of alcohol. The salt is deliquescent in moist air. Its aqueous solution is neutral to litmus.

When ignited the salt first yields oxygen with the formation of sodium nitrite; later, oxides of nitrogen are evolved. When heated with organic matter or reducing substances, for example sodium thiosulphate, oxidation takes place, sometimes with explosive violence.

Sodium nitrate is used principally as an oxidizing agent in the dry way, especially for the destruction of organic matter in the detection and determination of chlorine, sulphur, arsenic, etc., in organic substances; also for the detection of chromium and manganese and as a constituent of oxidizing fluxes.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0050%
Chlorides (Cl)	0.0050%
Chlorates and Perchlorates (as Cl)	0.0050%
Calcium (Ca)	0.0100%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%
Nitrites (N_2O_4)	0.0001%
Iodates (I_2O_5)	0.0020%

Methods of Testing

Sulphates.—Dissolve 3 gm. of sodium nitrate in 50 mls of water, add a few drops of hydrochloric acid and some barium chloride solution and allow to stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with a few drops of nitric acid and add silver nitrate solution. Not more than a slight opalescence should be produced.

Chlorates and Perchlorates.—Gently ignite 1 gm., dissolve the residue in 20 mls of water, acidulate the solution with nitric acid and add silver nitrate solution. Not more than a slight opalescence should result.

Calcium.—Dissolve 3 gm. of sodium nitrate in 50 mls of water and add a few mls of ammonia water and some ammonium oxalate solution. No change should be apparent.

Iron.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with hydrochloric acid and add potassium sulphocyanate solution. No red color should develop.

Other Heavy Metals.—Dissolve 1 gm. in 10 mls of water and add a few drops each of ammonia water and ammonium sulphide solution. No precipitate or dark color should result.

Nitrites and Iodates.—Dissolve 0.5 gm. in 10 mls of water and add 3 or 4 drops of diluted sulphuric acid and some zinc iodide-starch solution. No blue color should develop within one minute.

SODIUM NITRITE

NaNO_2

Mol. Wt. 69.01

Sodium nitrite is usually in the form of white or slightly yellowish, hygroscopic, cylindrical sticks. It is soluble in about 2 parts of water, forming a solution alkaline to litmus. It contains at least 97% of NaNO_2 .

Sodium nitrite is less hygroscopic than potassium nitrite, but when exposed to the air it gradually deliquesces and becomes oxidized to the nitrate. Its solutions are also unstable, undergoing oxidation on exposure.

The salt is largely used in organic synthesis and in preparing nitric oxide. It is also employed in the detection of phenols, alkaloids, diastase, naphthols, gurjun balsam and antipyrine and in the elaidin test.

The salt should be kept in tightly-stoppered containers.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Sulphate (SO_4)	0.0100%
Heavy Metals	0.0000%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 5 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with a few drops of hydrochloric acid and add barium chloride solution. No turbidity or opalescence should be produced.

Heavy Metals.—Dissolve 1 gm. of sodium nitrite and 1 gm. of ammonium chloride in 5 mls of water, evaporate to dryness, dissolve the residue in 10 mls of water and add hydrogen sulphide water. No change should be apparent. Add further a slight excess of ammonia water and a few drops of ammonium sulphide solution. No dark color or precipitate should be produced.

Quantitative Method.—Dissolve about 1 gm., accurately weighed, in water and dilute to 100 mls. Allow 15 mls of the solution to run slowly and with constant stirring into a mixture of 50 mls of tenth-normal potassium permanganate solution, 300 mls of water and 50 mls of 10% sulphuric acid. After ten minutes add to the mixture 3 gm. of potassium iodide and titrate the liberated iodine with tenth-normal sodium thiosulphate solution, using starch solution as indicator. At least 97% of NaNO_2 should be found.

1 ml of tenth-normal $\text{KMnO}_4 = 0.0034505$ gm. of NaNO_2 ,
log 53788.

SODIUM NITRITE, FREE FROM POTASSIUM

NaNO_2

Mol. Wt. 69.01

Sodium nitrite, free from potassium, has the physical properties described under Sodium Nitrite. It contains at least 97% of NaNO_2 . This reagent is especially useful in preparing sodium-cobaltic nitrite solution for the detection of potassium. It may also be used for the other purposes mentioned under Sodium Nitrite.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0100%
Heavy Metals	0.0000%
Potassium (K)	0.0200%

Methods of Testing

Chlorides, Sulphates and Heavy Metals.—Carry out the tests for these impurities as directed under Sodium Nitrite.

Potassium.—Dissolve 10 gm. of sodium nitrite and 4 gm. of cobalt chloride in 50 mls of water, acidulate the solution with 2 mls of 36% acetic acid, dilute to 100 mls and allow the mixture to stand at 40° to 50° C. for twenty-four to forty-eight hours. No yellow precipitate should form.

Quantitative Method.—Perform this test as directed under Sodium Nitrite. At least 97% of NaNO₂ should be found.

SODIUM-COBALTIC NITRITE SOLUTION

Sodium-cobaltic nitrite solution is a clear, reddish-brown liquid usually having a slight odor of nitric oxide. It may be prepared by dissolving 4 gm. of cobaltous chloride and 10 gm. of sodium nitrite in about 50 mls of water, adding 2 mls of 36% acetic acid and diluting to 100 mls. The cobaltous chloride may be substituted by cobaltous nitrate or acetate. The formula given above is official in the U. S. Pharmacopœia, 9th Revision.

Sodium-cobaltic nitrite solution is used principally for the detection of potassium, a yellow precipitate of potassium-cobaltic nitrite being formed in the presence of even minute amounts of potassium salts. Salts of ammonium, rubidium and caesium are likewise precipitated by the reagent.

Sodium-cobaltic nitrite solution deteriorates and should not be kept longer than three months. A few drops of

acetic acid should be added to the solution from time to time and any precipitate which may form on standing should be removed.

SODIUM NITROFERRICYANIDE

(Sodium Nitroprusside)

$\text{Na}_2\text{Fe}(\text{CN})_5\text{NO} + 2\text{H}_2\text{O}$ Mol. Wt. 297.96

Sodium nitroferricyanide is usually in the form of ruby-red, transparent crystals, clearly soluble in about 2.5 parts of water.

It is used in analysis for many purposes among which may be mentioned the detection of hydrogen sulphide and alkaline sulphides; of creatinine, cystin, aceto-acetic acid, acetone and aldehydes; the detection of coniin, strophanthin and other alkaloids and the determination of quinine; the detection of saccharin, paracresol and hydroxylamine.

Solutions of sodium nitroprusside undergo decomposition on keeping, a blue deposit forming. They should therefore be freshly prepared when used.

Maximum Limit of Impurity

Sulphates (SO_4) 0.0100%

Method of Testing

Sulphates.—Dissolve 1 gm. in 50 mls of water, add 1 mil of hydrochloric acid and some barium chloride solution. The liquid should not become turbid.

SODIUM OXALATE

(Soerensen's Oxalate)

$\text{Na}_2\text{C}_2\text{O}_4$ Mol. Wt. 134.01

Sodium oxalate is a white, crystalline powder, soluble in about 31 parts of cold, and in about 16 parts of boiling water. It contains at least 99.9% of $\text{Na}_2\text{C}_2\text{O}_4$.

Sodium oxalate is stable when heated to 240° to 250° C. and this temperature is necessary for expelling all traces of occluded moisture in the preparation of the reagent. Hygroscopic moisture is readily removed at 105° C. The salt, however, is hygroscopic to only a very slight degree. Solutions of sodium oxalate are stable on boiling but they readily attack glass, even when kept cool; for this reason it is inadvisable to use a stock solution as a volumetric standard.

The reagent is used as a starting material for preparing volumetric solutions for acidimetry and oxidimetry and it has been selected for this purpose by the Bureau of Standards of the U. S. Department of Commerce. Bureau of Standards Circular No. 40 (1913), entitled "Sodium Oxalate as a Standard in Volumetric Analysis," is the source from which the methods of testing this reagent have been adapted. The methods of the Bureau were in turn based upon the original experiments of Soerensen.*

The reagent should be stored in well-stoppered bottles and should be thoroughly dried at 105° C. before use.

Maximum Limits of Impurities

Hygroscopic Moisture	0.0100%
Sodium Bicarbonate (NaHCO_3)	0.0336%
Sodium Binoxalate (NaHC_2O_4)	0.0224%
Insoluble Matter	0.0100%
Sulphates (SO_4)	0.0010%
Chlorides (Cl)	0.0020%
Iron (Fe)	0.0002%
Potassium (K)	0.0600%
Organic Impurities	Trace

Methods of Testing

Hygroscopic Moisture.—Dry 10 gm. of the sodium oxalate at 105° C. to constant weight. The loss should not exceed 0.001 gm.

Sodium Bicarbonate, Sodium Binoxalate.—Prepare water free from carbon dioxide by boiling for some time

* *Zeit. Anal. Chem.* 42, 512 (1903); *abst. J. Chem. Soc.*, 84, II, 684, 750 (1903).

while passing through it a current of air free from carbon dioxide. Dissolve 2 gm. of the sodium oxalate in 150 mls of this prepared water in a flask of resistant glass, add 0.2 mil of phenolphthalein solution and boil the mixture for ten minutes while passing through it a current of air free from carbon dioxide. Prepare a color standard by adding 0.2 mil of phenolphthalein to 150 mls of water, containing 10 mls of tenth-normal sodium hydroxide solution, and diluting 6 mls of this red liquid to 100 mls with carbon dioxide-free water. Titrate the sodium oxalate solution with hundredth-normal acid or alkali as required to bring its color to match that of the prepared standard. Not more than 0.8 mil of hundredth-normal acid, or 0.4 mil of hundredth-normal alkali solution should be required.

Insoluble Matter.—Dissolve 10 gm. in 400 mls of water, filter off, dry and weigh any insoluble matter. The weight should not exceed 0.001 gm.

Sulphates.—Ignite 10 gm. to carbonate in a platinum crucible, using an alcohol burner to avoid contamination from a gas flame. Dissolve the residue in 50 mls of water, add hydrochloric acid until slightly acid, heat to boiling, add 5 mls of barium chloride solution and allow to stand twelve to eighteen hours. Any precipitate forming should not weigh more than 0.0005 gm.

Chlorides.—Ignite 10 gm. of the salt to carbonate, dissolve it in 75 mls of water, acidify the solution with nitric acid, filter if necessary, and add silver nitrate solution. Not more than a slight opalescence should be produced.

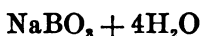
Iron and Potassium.—Ignite 10 gm. to carbonate and dissolve the residue in 50 mls of water. To one half the solution add hydrochloric acid to acidity and then potassium sulphocyanate solution. No red color should result. To the other half of the solution add acetic acid to acidity, then add sodium-cobaltic nitrite solution and allow it to stand three to four hours. Not more than a slight precipitate should form.

Organic Impurities.—Heat 1 gm. in a recently ignited test-tube with 10 mls of sulphuric acid until the appearance of fumes of sulphur trioxide. The acid should not acquire more than a faint brownish-tinge.

Quantitative Method.—Dry the salt to constant weight at 105° C., weigh out 0.2 to 0.3 gm. of the dried material, heat it cautiously in a covered platinum crucible until it is completely converted to carbonate (usually fifteen to thirty minutes), at the end of the operation partly removing the cover and applying sufficient heat to burn off all the carbon. The absorption of sulphur from the gas should be avoided either by using an alcohol lamp or by setting the crucible in a hole in an asbestos board. Dissolve the contents of the crucible in water and titrate the solution with tenth-normal hydrochloric acid solution, using methyl orange as indicator. At least 99.9% of $\text{Na}_2\text{C}_2\text{O}_4$ should be found.

1 mil of tenth-normal HCl = 0.0067005 gm. of $\text{Na}_2\text{C}_2\text{O}_4$,
log 82611.

SODIUM PERBORATE



Mol. Wt. 154.06

Sodium perborate usually occurs as white granules or a crystalline powder. It contains at least 9% of available O, corresponding to about 86.5% of $\text{NaBO}_3 + 4\text{H}_2\text{O}$.

When kept cool and dry the salt is stable, but decomposes on exposure to heat or moisture with liberation of oxygen. The aqueous solution when kept cold decomposes slowly with the formation of hydrogen peroxide and a metaborate and liberation of oxygen; heat hastens this change.

Sodium perborate is soluble in about 40 parts of water. Boric, tartaric or citric acid increases the solubility of the salt in water. The aqueous solution has essentially the properties of hydrogen peroxide, oxidizing sulphides, liberating iodine from iodides, reducing mercury and gold salts to the metallic state, etc. By introducing the salt into 50% sulphuric acid, boric acid separates and a very active solution of hydrogen peroxide may be obtained.

Sodium perborate is used chiefly as an oxidizing agent and in the detection of blood and boiled milk. It should be stored in tightly-stoppered bottles in a cool place.

Method of Testing

Quantitative Method.—Dissolve about 0.25 gm., accurately weighed, in a mixture of 50 mls of water and 10 mls of diluted sulphuric acid and titrate the solution with tenth-normal solution of potassium permanganate. At least 9% of available oxygen should be found.

1 mil of tenth-normal $\text{KMnO}_4 = 0.0008$ gm. of available O,
log 90309.

SODIUM PEROXIDE

(Sodium Superoxide)

Na_2O_2 ,

Mol. Wt. 78.0

Sodium peroxide is a yellowish, often somewhat granular, hygroscopic powder, easily soluble in water with evolution of oxygen, yielding a strongly alkaline solution. It contains at least 90% of Na_2O_2 .

Exposed to the air sodium peroxide takes up moisture and carbon dioxide, becoming gradually changed to sodium carbonate with the simultaneous liberation of oxygen and the acquisition of a white color. When dissolved in dilute acids hydrogen peroxide is formed. When brought in contact with organic matter, such as paper, straw, sawdust, etc., especially when moist, ignition, and sometimes explosion, is likely to take place. Heat or percussion tends to provoke this result.

Sodium peroxide is an energetic oxidizing agent; it also exerts a reducing action upon mercury, silver and gold salts, permanganates and some other compounds. As a reagent it is employed chiefly as an oxidizer in detecting and determining sulphur, nitrogen, phosphorus and halogens in organic and inorganic combination; for destroying organic matter; as a component of fluxes; and in the Parr calorimeter.

Sodium peroxide should be stored in air-tight containers, in a cool, dry place and must be very carefully protected from contact with organic matter.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0150%
Chlorides (Cl)	0.0033%
Phosphates (P_2O_5)	0.0060%
Silicates (SiO_2)	0.0100%
Iron (Fe)	0.0015%
Other Heavy Metals	0.0000%
Nitrogen Compounds (N)	0.0030%

Methods of Testing

Sulphates.—Add 5 gm. gradually to a mixture of 20 mls of hydrochloric acid and 100 mls of water, add barium chloride solution to the clear liquid and let it stand twelve to eighteen hours. No precipitate should form.

Chlorides.—Add 3 gm. gradually to a mixture of 20 mls of nitric acid and 100 mls of water and to the liquid add silver nitrate solution. Not more than a slight opalescence should develop.

Phosphates.—Add 2.5 gm. gradually to a mixture of 20 mls of nitric acid and 100 mls of water, to the solution add 25 mls of ammonium molybdate solution and allow it to stand 2 hours at about 40°C . No yellow precipitate should form.

Silicates.—Add 10 gm. gradually to a mixture of 40 mls of hydrochloric acid and 100 mls of water, evaporate the solution on the steam-bath, dry the residue at about 120°C . for half an hour and dissolve it in a mixture of 10 mls of hydrochloric acid and 90 mls of water. Filter off any insoluble matter and wash, ignite and weigh it. Its weight should not exceed 0.001 gm.

Iron.—Dissolve 1 gm. in 20 mls of water, add nitric acid in slight excess followed by solution of ammonium sulphocyanate. Not more than a slight reddish color should develop.

Other Heavy Metals.—Add 5 gm. cautiously to 100 mls of water. To 40 mls of the solution add 10 mls of hydrochloric acid followed by hydrogen sulphide water, and to another portion of 40 mls a few drops of ammonium sulphide solution. In neither case should a precipitate or brown color be visible, although in the latter a slight, greenish color may develop.

Nitrogen Compounds.—Mix 1 gm. with 0.3 gm. of dextrose, very cautiously, in a capacious nickel crucible. Deflagrate the mixture by heating the covered crucible very gently. Allow to cool and dissolve the contents of the crucible in 5 mls of water, acidulate with 10 mls of diluted sulphuric acid and overlay 10 mls of this solution on 5 mls of diphenylamine solution. No blue color should develop at the plane of contact.

In testing the reagent for nitrogen compounds by this method, the peroxide must be completely decomposed, as otherwise hydrogen peroxide may be present in the solution tested, which will then give a blue color with the diphenylamine solution. Thorough mixing of the peroxide and the dextrose is therefore advisable. The amount of dextrose specified is usually sufficient for complete reduction although it may be slightly exceeded if necessary.

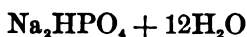
Quantitative Method.—Introduce about 0.5 gm., accurately weighed, into the outer compartment of the decomposition flask of a Lunge gas volumeter,* and in the inner put a mixture of 15 to 20 mls of diluted sulphuric and 2 or 3 drops of saturated solution of cobalt nitrate. When the apparatus is properly adjusted, tilt the flask to allow the acid mixture to act upon the sodium peroxide, and measure the oxygen evolved.

1 mil of O at 0° C. and 760 mm. = 0.006964 gm. of Na_2O_2 ,
log 84286.

* For an illustration of this apparatus see Olsen's *Quantitative Analysis*, 5th ed., 317 (1919).

SODIUM PHOSPHATE

(Sodium Phosphate, Dibasic; Secondary Sodium Phosphate)



Mol. Wt. 358.24

Sodium phosphate occurs usually as colorless crystals or granules, soluble in about 6 parts of water. The salt is efflorescent in dry air and its aqueous solution is alkaline to litmus paper.

When heated the salt melts at about 35° C. and at 100° C. it is rendered anhydrous; the anhydrous salt is hygroscopic. On ignition dibasic sodium phosphate is changed to sodium pyrophosphate; this affords an easy approximate method of assaying the salt or its solution in the absence of other salts.

Sodium phosphate is used chiefly in the detection of the alkaline earths and in the determination of magnesium and zinc; in standardizing uranium solutions; differentiating cinchona alkaloids; and in the recovery of molybdic acid.

The reagent should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Carbonates	0.0000%
Sulphates (SO ₃)	0.0100%
Chlorides (Cl)	0.0050%
Nitrates (N ₂ O ₅)	0.0016%
Heavy Metals	0.0000%
Arsenic (As)	0.0005%
Potassium Salts	Trace

Methods of Testing

Carbonates.—Add hydrochloric acid to a 10% solution of the salt. No effervescence should be visible.

Sulphates.—Dissolve 1 gm. in 20 mls of water, acidulate the solution with 1 mil of hydrochloric acid and add barium chloride solution. No precipitate should form.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add 2 mls of nitric acid and some silver nitrate solution. Not more than a slight opalescence should be produced.

Nitrates.—Dissolve 2 gm. in 10 mls of water, add a few milligrams of sodium chloride, a drop of indigo solution and 10 mls of sulphuric acid and shake the mixture. The blue color should not disappear at once.

Heavy Metals.—Dissolve 2 gm. in 20 mls of water, add 1 ml of hydrochloric acid and some hydrogen sulphide water. No change should be apparent. Then add 5 mls of ammonia water and a few drops of ammonium sulphide solution. No precipitate or dark color should result.

Arsenic.—Dissolve 2 gm. in 50 mls of water, introduce the solution a little at a time into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

Potassium Salts.—Introduce the salt into a non-luminous flame and view it through a cobalt glass. Not more than a slight, transient, violet color should be visible.

SODIUM PHOSPHATE, DIBASIC, ANHYDROUS

(Soerensen's Sodium Phosphate)

Na_2HPO_4

Mol. Wt. 142.05

Sodium phosphate, dibasic, anhydrous, is a white, hygroscopic powder, soluble in about 10 parts of water. Its solutions have an alkaline reaction to litmus. The salt may contain at most 0.1% of moisture.

The salt which has taken up an excess of moisture by undue exposure to the air may again be rendered anhydrous by drying at 100° to 150° C. Too high a temperature should be avoided as the change to pyrophosphate begins somewhat above 200° C.

The water used as a solvent in applying the tests under the methods of testing given below should be as free from carbon dioxide as possible. It may be prepared by the process described under Potassium Phosphate, Monobasic, Anhydrous.

This reagent is intended especially for use in connection with Potassium Phosphate, Monobasic, Anhydrous. in de-

termining the hydrogen ion concentration of bacteriological culture media and other fluids. Regarding the subject see Clark and Lubs, *The Colorimetric Determination of Hydrogen Ion Concentration and its Applications in Bacteriology*, Jour. of Bacteriology, 2, 1, 109, 191 (1917).

The salt should be preserved in tightly-stoppered bottles.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Sulphates (SO ₃)	0.0050%
Chlorides (Cl)	0.0020%
Moisture	0.1000%

Methods of Testing*

Insoluble Matter.—Dissolve 5 gm. in 100 mils of water. The solution should be free from insoluble matter.

Sulphates.—Dissolve 5 gm. in 100 mils of water, add 5 mils each of hydrochloric acid and barium chloride solution and allow it to stand twelve to eighteen hours. No precipitate of barium sulphate should form.

Chlorides.—Dissolve 5 gm. in 100 mils of water, add 5 mils of nitric acid, sp. gr. 1.40–1.42, and some silver nitrate solution. Not more than a slight opalescence should be produced.

Moisture.—Dry about 2 gm. of the sodium phosphate, accurately weighed, at 100° C. to constant weight. The loss in weight should not exceed 0.1%.

Loss on Ignition (Proof of Proper Composition).—Dry the salt to constant weight at 100° C., weigh out accurately about 2 gm. of the dried material and ignite it carefully to constant weight. The loss on ignition should be not less than 6.24% nor more than 6.44%.

Test for Proper Hydrogen Ion Concentration.—For this test the following special reagents are required:

* These methods of testing are adapted from Soerensen's original article, *Ergebnisse der Physiologie*, 12, 433 (1912).

Boric Acid. This should conform to the following tests of purity: (a) Heat a mixture of 20 gm. of the boric acid and 100 mls of water on a steam-bath for about fifteen minutes, shaking frequently. Cool the solution in ice-water and filter. Ten-mil portions of the filtrate should not be changed in appearance by the addition of barium chloride or silver nitrate solution. (b) Add 2 drops of methyl orange solution to each of three 5-mil portions of the filtrate obtained under (a). To one add 1 drop of tenth-normal sodium hydroxide solution. The color should change to yellow. To another add 1 drop of tenth-normal hydrochloric acid solution. A pronounced reddish tint should be produced. The third tube serves as a control. (c) Evaporate to dryness in a weighed platinum capsule 20 mls of the filtrate obtained under (a). Treat the residue with about 10 mls of hydrofluoric acid and 5 mls of sulphuric acid, evaporate the mixture and ignite and weigh any residue remaining. The weight should not exceed 0.002 gm.

Sodium Borate Solution. Dissolve 12.404 gm. of the boric acid described in the preceding paragraph in 100 mls of normal sodium hydroxide solution, free from carbonate, and dilute with water to 1 liter.

Phenolphthalein Solution. Dissolve 0.5 gm. of phenolphthalein in 1 liter of 50% alcohol.

The test is performed as follows: Dissolve 0.957 gm. of the sodium phosphate in water and dilute to 100 mls. Place in test-tubes three 10-mil portions of the solution, and for comparison use five other tubes containing the following mixture:

No. 1—8 mls of sodium borate solution + 2 mls of tenth-normal hydrochloric acid solution.

No. 2—9 mls of sodium borate solution + 1 mil of tenth-normal hydrochloric acid solution.

No. 3—10 mls of sodium borate solution.

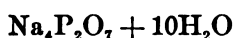
No. 4—9 mls of sodium borate solution + 1 mil of tenth-normal sodium hydroxide solution.

No. 5—8 mls of sodium borate solution + 2 mls of tenth-normal sodium hydroxide solution.

Add to each of the eight tubes 5 drops of the phenolphthalein solution and compare the colors of the liquids. The color of the tubes of sodium phosphate solution should lie between those of No. 2 and No. 4. The addition of 1 drop of tenth-normal hydrochloric acid solution to one of the tubes of phosphate solution and of 1 drop of tenth-normal sodium hydroxide solution to another should shift the color beyond the boundaries established by control tubes Nos. 2 and 4.

SODIUM PYROPHOSPHATE

(Tetra-sodium Pyrophosphate)



Mol. Wt. 446.24

Sodium pyrophosphate is usually in the form of colorless crystals, efflorescent in dry air and soluble in about 10 parts of water. Its aqueous solution is alkaline to litmus. When its solution is boiled in the presence of acetic or a mineral acid the salt quickly changes to an orthophosphate.

Sodium pyrophosphate is used chiefly in the electrolytic separation of various metals. It should be stored in tightly-stoppered bottles.

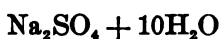
Maximum Limits of Impurities

Orthophosphates (as P_2O_5)	1.0000%
Carbonates	0.0000%
Sulphates (SO_4)	0.0100%
Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0016%
Heavy Metals	0.0000%
Arsenic (As)	0.0005%
Potassium Salts	Trace

Methods of Testing

Orthophosphates.—Dissolve 2 gm. in 20 mls of water and add silver nitrate solution. The precipitate should be pure white.

Other Tests.—Perform the tests directed under Sodium Phosphate. The results should be as there indicated.

SODIUM SULPHATE, CRYSTALS

Mol. Wt. 322.22

This form of sodium sulphate occurs usually as moderately large, colorless, efflorescent crystals; sometimes in the form of small needles or granules. It is soluble in about 3 parts of water, but is insoluble in alcohol. The aqueous solution is neutral to litmus.

The salt effloresces very easily and if left exposed to the air will gradually lose all its water of crystallization. At about 33° C. the salt melts, but on further heating loses its water and again becomes solid. Anhydrous sodium sulphate may be heated moderately without change and upon this behavior is based the determination of sodium in some of its organic salts by ignition with sulphuric acid, sodium sulphate resulting. At white heat the salt volatilizes.

The reagent is employed chiefly as a precipitant in the detection and determination of barium and lead; in removing lead and bismuth from solutions to be polarized or otherwise treated; and in the determination of fusel oil.

Sodium sulphate, crystals, should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Substances Insoluble in Water	0.0000%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%
Chlorides (Cl)	0.0010%
Calcium (Ca)	0.0200%
Magnesium (Mg)	0.0050%
Arsenic (As)	0.0005%

Methods of Testing

Substances Insoluble in Water.—Dissolve 5 gm. in 50 mls of water. No insoluble matter should remain.

Iron.—Dissolve 1 gm. in 20 mls of water, add a few drops of nitric acid, heat to boiling and add potassium sulphocyanate solution. No red color should be produced.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add silver nitrate solution. No change should be apparent.

Calcium and Magnesium.—Dissolve 1 gm. in 20 mls of water, add a few mls of ammonia water and then ammonium oxalate and ammonium phosphate solutions. No apparent change should be produced by either reagent.

Arsenic.—Dissolve 2 gm. in 20 mls of water, introduce the solution into a Marsh apparatus and allow the action to proceed for one hour. No deposit of arsenic should be visible in the reduction tube.

SODIUM SULPHATE, ANHYDROUS

Na_2SO_4

Mol. Wt. 142.06

Anhydrous sodium sulphate is a white powder, readily soluble in about 7 parts of water; insoluble in alcohol. Its 10% solution is neutral to litmus.

The reagent is chiefly used in the Kjeldahl nitrogen determination, as a dehydrating agent for organic fluids and for the ordinary purposes for which Sodium Sulphate, Crystals, is employed.

Maximum Limits of Impurities

Insoluble Matter	0.0000%
Water	0.5000%
Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0032%
Ammonium Compounds (NH_3)	0.0050%
Iron (Fe)	0.0008%
Other Heavy Metals	0.0000%

Methods of Testing

Insoluble Matter.—5 gm. of the salt should dissolve in 100 mls of water without residue.

Water.—Gently ignite the salt. The loss in weight should not exceed 0.5%.

Chlorides.—Dissolve 1 gm. in 20 mls of water and add a few drops each of nitric acid and silver nitrate solution. Not more than a slight opalescence should be produced.

Nitrates.—Dissolve 1 gm. in 20 mls of water, add a few milligrams of sodium chloride, a drop of indigo solution and 10 mls of sulphuric acid and shake the mixture. The blue color should not disappear.

Ammonium Salts.—Dissolve 1 gm. in 25 mls of water and add 0.5 ml of Nessler's solution. At most a slight yellow color should result.

Iron.—Dissolve 1 gm. in 20 mls of water, boil the solution with a few drops of nitric acid and add potassium sulphocyanate solution. No red color should be produced.

Other Heavy Metals.—Dissolve 1 gm. in 20 mls of water and add hydrogen sulphide water. No change should be apparent.

SODIUM SULPHIDE

$\text{Na}_2\text{S} + 9\text{H}_2\text{O}$

Mol. Wt. 240.2

Sodium sulphide is usually in the form of clear, colorless, deliquescent crystals, easily and clearly soluble in water (about 2 parts). The aqueous solution is alkaline to litmus. The reagent contains at least 95% of $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$.

Exposed to the air the salt takes up moisture and also gradually oxidizes to the sulphate. Its solutions change slowly in the air with the formation of thiosulphate and carbonate; but when protected from the air by a layer of oil or similar material, solutions keep well.

Sodium sulphide is used chiefly for the precipitation and separation of copper, bismuth, cadmium, antimony and tin; for determining copper and zinc; in the determination of iodine in mixtures; and in the Kjeldahl nitrogen method for removal of mercury before distillation.

The reagent should be kept in well-closed, glass-stoppered bottles.

Maximum Limits of Impurities

Nitrogen (N)	0.0070%
Sulphites and Thiosulphates (as S)	0.3200%

Methods of Testing

Nitrogen.—Dissolve 10 gm. of sodium sulphide in 100 mls of water in a distillation flask, add 1 gm. each of zinc dust and iron by hydrogen (or 2 gm. of Devarda's metal) and 50 mls of 27% sodium hydroxide solution and connect the flask with a bulb-tube, a condenser and a receiver containing 10 mls of tenth-normal hydrochloric acid solution. Distil off about 50 mls and titrate the distillate with tenth-normal sodium hydroxide solution, using methyl orange as indicator. At least 9.5 mls of the alkali solution should be required to effect neutrality.

Sulphites and Thiosulphates.—Dissolve 1 gm. in 100 mls of water, add 2 gm. of zinc sulphate dissolved in 150 mls of water, shake the mixture vigorously and allow it to stand for half an hour. Filter and titrate 50 mls of the filtrate with tenth-normal iodine solution, using starch solution as indicator. Not more than 0.5 ml should be required.

Quantitative Method.—Weigh accurately about 0.3 gm. of the salt and rinse it into a flask containing 40 mls of tenth-normal iodine solution. When the sulphide has entirely dissolved add 2 to 3 mls of hydrochloric acid and titrate the excess iodine with tenth-normal sodium thiosulphate solution. At least 95% of $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$ should be found.

1 ml of tenth-normal I = 0.01201 gm. of $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$,
log 07954.

SODIUM SULPHIDE SOLUTION, 5%

Sodium sulphide solution is a clear, colorless or slightly yellowish liquid, containing from 4.5% to 5.5% of Na_2S .

The solution is used chiefly in the Kjeldahl nitrogen determination and should be kept in well-closed, rubber-stoppered bottles, containing little air space.

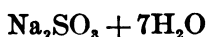
Maximum Limit of Impurity

Nitrogen 0.0010%

Methods of Testing

Nitrogen.—Perform the test as given under Sodium Sulphide, using 100 mls of sodium sulphide solution, 5%. Not more than 0.75 mil of tenth-normal solution of hydrochloric acid should be neutralized by the distillate.

Quantitative Method.—Dilute about 5 mls, accurately weighed, to 100 mls in a graduated flask. Allow 25 mls of this solution to run into 40 mls of tenth-normal iodine solution to which have been added 2 or 3 mls of hydrochloric acid, and titrate the excess iodine with tenth-normal sodium thiosulphate solution. From 4.5% to 5.5% of Na_2S should be found in the original solution of sodium sulphide. 1 mil of tenth-normal $\text{I} = 0.003903$ gm. of Na_2S , log 59140.

SODIUM SULPHITE

Mol. Wt. 252.17

Sodium sulphite is usually in the form of colorless, efflorescent crystals, soluble in about 3 parts of water, forming solutions alkaline to litmus. It contains at least 94% of $\text{Na}_2\text{SO}_3 + 7\text{H}_2\text{O}$. On exposure to air the salt readily loses its water of crystallization and also gradually oxidizes to the sulphate. It oxidizes also in solution, more quickly in dilute than in concentrated condition.

Sodium sulphite is used principally as a reducing agent and in the assay of certain aldehydes and ketones. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl) 0.0100%
 Heavy Metals Trace
 Arsenic (As) 0.0005%

Methods of Testing

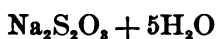
Chlorides, Heavy Metals and Arsenic.—Perform the tests as directed under Potassium Bisulphite, *Meta*. The results should be as there stated.

Quantitative Method.—Dissolve 0.25 to 0.5 gm. of the salt, accurately weighed, in 50 mls of tenth-normal iodine solution, acidulate with 5 mls of hydrochloric acid and titrate the excess iodine with tenth-normal sodium thiosulphate solution. At least 94% of $\text{Na}_2\text{SO}_3 + 7\text{H}_2\text{O}$ should be found.

1 mil of tenth-normal I = 0.0126085 gm. of $\text{Na}_2\text{SO}_3 + 7\text{H}_2\text{O}$, log 10067.

SODIUM THIOSULPHATE

(Sodium Hyposulphite)



Mol. Wt. 248.2

Sodium thiosulphate is usually in the form of colorless crystals, efflorescent in warm, dry air and slightly deliquescent in moist air. It is soluble in less than 1 part of water, but insoluble in alcohol. Its aqueous solution is neutral or slightly alkaline to litmus. The salt contains at least 99% of $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$.

When heated the crystals melt at about 45° to 50° C. and at about 100° C. the salt begins to decompose. Aqueous solutions gradually deposit sulphur with the formation of sulphite and finally, on exposure to air, of sulphate. Acidulation of the solution with hydrochloric or sulphuric acid liberates sulphur dioxide and a white precipitate of sulphur also forms. The production of this precipitate serves to distinguish the thiosulphate from the sulphite or bisulphite, neither of which yields a precipitate on acidulation.

The reagent is used, chiefly in the form of its volumetric solution, in the determination of the halogens, hypochlorites, iron salts, chromic acid and chromates, manganese dioxide, lead dioxide, permanganates, peroxides and copper salts; for estimating pentavalent vanadium and determining sulphur in illuminating gas; and as a reagent for aldehydes, hydrocyanic acid, quinine, hydrated chloral, carbon monoxide and alkaloids.

Sodium thiosulphate should be stored in well-stoppered bottles in a cool place.

Maximum Limits of Impurities

Free Alkali (as NaOH)	0.0100%
Sulphides (S)	0.0130%
Calcium (Ca)	0.0200%

Methods of Testing

Free Alkali.—Dissolve 1 gm. in 10 mls of water and add phenolphthalein solution. No red color should develop.

Sulphides.—Dissolve 1 gm. in 10 mls of water and add zinc sulphate solution. No change should be apparent.

Calcium.—Dissolve 1 gm. in 20 mls of water and add ammonia water and ammonium oxalate solution. No turbidity should result.

Quantitative Method.—Dissolve the salt in water and titrate the solution with standard iodine solution, using starch as indicator. At least 99% of $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$ should be found.

1 mil of tenth-normal I = 0.02482 gm. of $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$,
log 39480.

SODIUM TUNGSTATE

Mol. Wt. 330.03

Sodium tungstate is usually in the form of colorless crystals or white crystalline lumps or powder. It is soluble in about 4 parts of water. The salt contains not more than 12% of water (theory 10.91%) and not less than 69% of WO_3 (theory 70.29%).

Sodium tungstate in solution is easily decomposed by acids, especially by strong mineral acids and even by acetic, citric, or boric acid, and by carbon dioxide. By the action of the latter, sodium paratungstate ($\text{Na}_{10}\text{W}_{12}\text{O}_{41} + 28\text{H}_2\text{O}$) is formed, small amounts of which are nearly always present in sodium tungstate.

The reagent is used chiefly for preparing phosphotungstates and borotungstates and in reagents for alkaloids, glucose, tannin, blood, peptone, bile pigments and acetoacetic acid.

Maximum Limits of Impurities

Chlorides (Cl)	0.0050%
Sulphates (SO ₄)	0.0750%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water, add 5 mls of nitric acid, filter and to the filtrate add a few drops of silver nitrate solution. Not more than a slight opalescence should be produced.

Sulphates.—Dissolve 1 gm. in 25 mls of water, add 5 mls of nitric acid, boil for ten or fifteen minutes, replacing the water lost by evaporation, filter and to the filtrate add barium nitrate solution. No turbidity should be produced at once.

Quantitative Determination of Water and Tungstic Acid (Anhydride).—Gently ignite about 1 gm. of sodium tungstate, accurately weighed. The loss in weight should not exceed 12%.

Dissolve about 1 gm., accurately weighed, in 10 mls of water, add 10 mls of hydrochloric acid, evaporate on the steam-bath to dryness and heat the residue half an hour at 120° C. Treat the residue with 20 mls of hydrochloric acid and repeat the entire operation three or four times. Finally digest the residue with ammonium nitrate solution to which a little nitric acid has been added, filter, wash the insoluble tungstic acid with dilute nitric acid, dry, ignite and weigh it as WO₃. At least 69% of WO₃ should be found.

SODIUM AND AMMONIUM PHOSPHATE**(Microcosmic Salt)**

Mol. Wt. 209.15

Sodium and ammonium phosphate usually occurs as colorless crystals or granules, soluble in about 5 parts of water and yielding a solution alkaline to litmus. The salt effloresces in the air with the loss of some ammonia. When slowly heated it melts easily (at about 80° C.), loses water

and ammonia, and changes to monobasic sodium phosphate, NaH_2PO_4 ; this on further heating changes to sodium metaphosphate, in which form it serves as a blow-pipe reagent for the identification of silica and various metals by the colors of their beads.

Besides its employment in blow-pipe analysis, sodium and ammonium phosphate may also be employed for many purposes in place of sodium phosphate, as for example, the precipitation of magnesium and zinc and standardizing uranium solutions.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Carbonates	0.0000%
Sulphates (SO_4)	0.0100%
Chlorides (Cl)	0.0050%
Nitrates (N_2O_5)	0.0016%
Heavy Metals	0.0000%
Arsenic (As)	0.0005%
Potassium Salts	Trace

Methods of Testing

Perform the tests as directed under Sodium Phosphate. The results should be as there stated.

THYMOL

$\text{C}_6\text{H}_3(\text{CH}_3)(\text{OH})(\text{C}_3\text{H}_7)1:3:4$ Mol. Wt. 150.16

Thymol occurs usually as colorless, translucent crystals, having an aromatic odor. It is soluble in about 1100 parts of water, but easily soluble in alcohol, ether or chloroform. Thymol melts at about 50°C . (theory 49.65°C . corr.), volatilizes readily on the steam-bath and boils at about 230°C . (theory 231.8°C .).

Thymol is used chiefly in the detection of sugars, coniferin, lignin, chloroform, iodoform, and bromine and in the colorimetric determination of titanium. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Inorganic Matter	0.0500%
Free acids	0.0000%
Phenol	0.0000%

Methods of Testing

Inorganic Matter.—Heat 2 gm. of thymol on the steam-bath and weigh any non-volatile residue. The weight should not exceed 0.001 gm.

Free Acids.—Blue litmus paper should not be reddened by aqueous or alcoholic solutions of thymol.

Phenol.—Dissolve 0.5 gm. in 10 mls of alcohol and add 1 drop of ferric chloride solution. A violet color should not be produced.

TIN

Sn

At. Wt. 118.7

Tin is a soft, almost silver-white metal, usually occurring as irregular granules, mossy pieces or coarse powder. It melts when pure at 232° C. In hot hydrochloric acid it dissolves with the formation of stannous chloride. Hot nitric acid converts it into insoluble metastannic acid.

Tin is used chiefly as a reducing agent; in separating phosphoric acid from the alkaline earths; in preparing stannous chloride; and for preparing standard tin solutions used in tin determinations by the iodine method.

Maximum Limits of Impurities

Lead (Pb)	0.0137%
Copper (Cu)	0.0004%
Iron (Fe)	0.0170%
Zinc (Zn)	0.0200%
Antimony	Trace
Arsenic	Trace

Methods of Testing

Lead.—Digest 5 gm. with 40 mls of nitric acid, sp. gr. 1.40–1.42, on a steam-bath until the metal is entirely converted into a white powder. Evaporate to dryness, stir the residue with 10 mls of nitric acid, sp. gr. 1.20, and

50 mils of water and filter. To the filtrate add 1 mil of diluted sulphuric acid, evaporate on the steam-bath as far as possible and then take up the residue with 10 mils of water. Collect, ignite and weigh any insoluble lead sulphate. The weight should not exceed 0.001 gm.

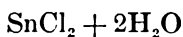
Copper, Iron and Zinc.—To the filtrate from the lead sulphate obtained in the preceding test add ammonia water until alkaline. The liquid should not acquire a blue color. Then add ammonium sulphide and allow it to stand at about 50° C. for four to five hours. Collect any precipitate which may form and ignite and weigh it. The weight should not exceed 0.003 gm.

Antimony.—Heat on the steam-bath 5 gm. of the tin and 50 mils of hydrochloric acid, adding potassium chlorate in small portions until solution results. Heat the solution nearly to boiling and add bright piano-wire until the latter ceases to dissolve. From this treatment no precipitate should result, or at most only a very slight black precipitate. The piano-wire used in this test should be soluble without residue in hydrochloric acid.

Arsenic.—Digest 5 gm. on the steam-bath with 40 mils of nitric acid, sp. gr. 1.40–1.42, until the metal is completely changed to a white powder. Evaporate completely, treat the residue with 10 mils of nitric acid, sp. gr. 1.20, and 50 mils of water and filter. Mix the filtrate with 5 mils of sulphuric acid and evaporate, first on the steam-bath, then on a sand-bath, until fumes of sulphuric acid begin to be evolved. Cool, dissolve the residue in 50 mils of water, introduce the solution gradually into a Marsh apparatus, and allow the action to proceed for one hour. Not more than a slight deposit should be visible in the reduction tube.

TIN CHLORIDE

(Stannous Chloride)



Mol. Wt. 225.65

Tin chloride is usually in the form of colorless crystals. It is soluble in less than 1 part of water, a little hydro-

chloric acid generally being required to hold it in solution; readily soluble in alcohol. Much water decomposes the salt with the formation of insoluble basic chloride. The reagent contains at least 95% of $\text{SnCl}_2 + 2\text{H}_2\text{O}$.

Tartaric acid or ammonium chloride, as well as hydrochloric acid, assist in making a clear aqueous solution of tin chloride. Its solution when exposed to the air gradually absorbs oxygen, forming oxychloride. To prevent this change and preserve the solution pieces of metallic tin are kept in contact with it.

Stannous chloride is an active and widely used reducing agent and to this property is largely due the employment of the salt in both qualitative and quantitative analysis. It is used chiefly in the detection of mercury, arsenic, cobalt, gold and bismuth; in the determination of copper and iron; in organic synthesis; and as a reagent for picric acid, morphine and glucose.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.1250%
Ammonium Compounds (NH_3)	0.0035%
Earths and Alkalis	0.1000%
Iron (Fe)	0.0001%
Arsenic (As)	0.00075%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 5 mls of hydrochloric acid, dilute the solution with 50 mls of water and add barium chloride solution. No change should be apparent.

Ammonium Compounds.—Heat 1 gm. with 10 mls of 27% sodium hydroxide solution and test the vapors with moist red litmus paper. The paper should not turn blue.

Earths, Alkalis and Iron.—Dissolve 2 gm. in 10 mls of hydrochloric acid, dilute with 100 mls of water and pass in hydrogen sulphide gas until the tin is all precipitated. Filter, evaporate the filtrate and ignite and weigh the residue. The weight should not exceed 0.002 gm.

Warm the ignited residue with 1 mil of hydrochloric acid, dilute with 20 mls of water and add potassium sulphocyanate solution. Not more than a slight red color should result.

Arsenic.—Boil 2 gm. with 10 mls of hydrochloric acid for a few minutes and allow the solution to stand one hour. It should remain clear and colorless.

Quantitative Method.—Dissolve about 0.5 gm., accurately weighed, in 2 mls of hydrochloric acid, dilute the solution with 50 mls of water, add 5 gm. of potassium and sodium tartrate and, when it has dissolved, sodium bicarbonate until the solution is alkaline to litmus paper. Then titrate with tenth-normal iodine solution, using starch solution as indicator. At least 95% of $\text{SnCl}_2 + 2\text{H}_2\text{O}$ should be found.

1 mil of tenth-normal I = 0.0112825 gm. of
 $\text{SnCl}_2 + 2\text{H}_2\text{O}$, log 05241.

TIN CHLORIDE SOLUTION

(Stannous Chloride Solution)

Tin chloride solution is a saturated solution of stannous chloride in concentrated hydrochloric acid. It is a clear, colorless or slightly yellow liquid having a specific gravity of at least 1.90.

The solution is used for detecting arsenic, especially in the presence of bismuth, antimony and other substances which interfere with the application of other arsenic tests. In applying the stannous chloride test it is necessary that nitrates, sulphates, sulphites, sulphides, and compounds of mercury, gold and selenium be absent.

The reagent should be stored in well-closed, glass-stoppered bottles and should be exposed to the air as little as possible to prevent oxidation.

Maximum Limits of Impurities

Substances Precipitated by Alcohol	0.0000%
Sulphates (SO_4)	0.0020%

Methods of Testing

Substances Precipitated by Alcohol.—Mix the solution with 10 volumes of alcohol and allow the mixture to stand for half an hour. No turbidity should result.

Sulphates.—To 5 mls of the solution add 50 mls of water and some barium nitrate solution and allow it to stand ten minutes. No turbidity should be produced.

URANIUM ACETATE, FREE FROM SODIUM
(Uranyl Acetate)



Uranium acetate is a yellow, crystalline powder having a slightly acetous odor. It is easily soluble in water although the addition of a little acetic acid is usually necessary to effect complete solution on account of the almost invariable presence of basic salt.

Uranium compounds are notably radio-active, fluorescent and luminescent. They are quite sensitive to light and act as catalysts in processes involving the use of sunlight.

Uranium acetate is used principally in the preparation of volumetric solutions; for detecting sodium microscopically; in the determination of arsenic, phosphorus, strontium, vanadium and bismuth; in the separation of chromium and vanadium; and as an activator in bacterial oxidation processes.

The reagent should be protected from light by storage in well-stoppered, amber-colored bottles.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0100%
Sodium (Na)	0.0460%
Earths (as Ca)	0.0050%
Uranous Salt (Uiv)	0.2386%
Foreign Metals	0.0000%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 20 mls of water, acidulated with 2 or 3 mls of 36% acetic acid, and add barium chloride solution. No reaction should be apparent.

Sodium.—Dissolve 5 gm. in 200 mls of water with the aid of 10 mls of 36% acetic acid, heat the solution to boiling, add an excess of ammonia water, filter and evaporate the filtrate to dryness. Ignite the residue, dissolve it in water and titrate the solution with normal hydrochloric acid solution, using methyl orange as indicator. Not more than 0.1 ml of the normal acid solution should be required to obtain the end-point.

Earths.—Dissolve 1 gm. in 20 mls of water, acidulated with 2 or 3 mls of 36% acetic acid, and add ammonia water until a permanent precipitate is produced, followed by ammonium carbonate solution until the precipitate is redissolved. No cloudiness or precipitate should then form within one minute.

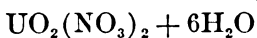
Uranous Salt.—Dissolve 1 gm. in 20 mls of water, acidulated with 2 mls of 10% sulphuric acid, and add 0.2 ml of tenth-normal potassium permanganate solution. A pink color should persist for a short time.

Foreign Metals.—Dissolve 5 gm. in 100 mls of water, acidulated with 5 mls of hydrochloric acid, heat the solution to boiling and pass into it hydrogen sulphide gas. No dark color or turbidity should appear within one minute.

Dissolve 1 gm. in 20 mls of water, acidulated with 2 mls of 36% acetic acid, add ammonia water until a permanent precipitate is produced, then ammonium carbonate until the precipitate is redissolved, and finally 2 or 3 drops of ammonium sulphide solution. No precipitate or dark brown color should be produced at once.

URANIUM NITRATE

(Uranyl Nitrate)



Mol. Wt. 502.32

Uranium nitrate is usually obtained as yellow, odorless crystals, having a greenish luster by reflected light. It is soluble in about 1 part of water, forming a solution acid to litmus; also readily soluble in alcohol and in ether. The

salt is somewhat efflorescent. It has the properties ascribed to uranium compounds under Uranium Acetate, Free From Sodium.

The salt is chiefly employed for the preparation of volumetric solutions; for the detection of hydrogen peroxide, phenols, mercury oxycyanide, morphine and other alkaloids; in the determination of phosphorus and arsenic; in separating tungstic acid from tungstates; and detecting cinnamic acid in benzoic acid.

Uranium nitrate should be stored in well-stoppered bottles of amber-colored glass.

Maximum Limits of Impurities

Sulphates (SO_4)	0.0050%
Alkali Salts	0.0500%
Earths (as Ca)	0.0050%
Uranous Salt (U^{IV})	0.1193%
Foreign Metals	0.0000%

Methods of Testing

Sulphates.—Dissolve 1 gm. in 20 mls of water and add barium chloride solution. No turbidity should develop within fifteen minutes.

Alkali Salts.—Ignite 1 gm. of the salt, powder the residue, ignite again and treat the residue with 20 mls of water. Filter, evaporate the filtrate to dryness and weigh the residue. The weight should not exceed 0.0005 gm.

Earths.—Dissolve 1 gm. in 20 mls of water, add ammonia water until a permanent precipitate is formed and then add ammonium carbonate solution until the precipitate redissolves. The solution should then remain clear for at least one minute.

Uranous Salt.—Dissolve 1 gm. in 20 mls of water and add 1 mil of 10% sulphuric acid and 0.1 mil of tenth-normal solution of potassium permanganate. The liquid should retain a pink color.

Foreign Metals.—Dissolve 5 gm. in 100 mls of water, add 5 mls of hydrochloric acid, heat the solution to boiling

and pass into it hydrogen sulphide gas. No dark color or precipitate should appear within one minute.

Dissolve 1 gm. in 20 mls of water, add ammonia water to the production of a permanent precipitate and then ammonium carbonate solution until the precipitate is redissolved. Then add 2 or 3 drops of ammonium sulphide solution. No dark brown color or precipitate should be produced at once.

ZINC

Zn

At. Wt. 65.37

Zinc is a moderately hard, lustrous, bluish-white metal. It is soluble in diluted hydrochloric or sulphuric acid and in solutions of the fixed alkali hydroxides.

The solution of pure zinc in acids takes place slowly but is hastened by the presence of other metals, such as gold, platinum, copper, cadmium, iron, etc., and a metal is often added for that purpose when a refractory zinc is being used. An impure zinc dissolves more readily than the pure metal.

When in dry condition zinc is not affected by exposure to the air but when moisture is present it gradually becomes coated with a layer of basic zinc carbonate. When heated, pure zinc melts at 419° C. and boils at 918° C. In the absence of air it can be distilled, but when ignited in the air it is changed to zinc oxide.

Zinc is obtainable in various forms such as sticks, granules, filings, coarse powder, fine powder (zinc dust) and the so-called "mossy" form, pieces of irregular shape with a large surface area. For various purposes several grades of zinc are employed, differing from each other in degree of purity.

Zinc is used principally in forensic analysis, especially in the detection and determination of arsenic; as a reducing agent in the determination of iron, copper, lead, molybdenum, uranium, phosphorus, and vanadic and nitric acids; for precipitating gold, platinum, iridium, nickel and cobalt; for preparing hydrogen; as a reagent for iodates, chlorates and nitrates; and for reducing various organic compounds.

Zinc should be stored in well-stoppered bottles. Prolonged exposure to the air should be avoided, especially with the finely divided metal. The collection of dust upon the zinc, due to lack of protection in storage, is likely to lead to erroneous results in exacting processes of analysis, as for example, the detection of small amounts of arsenic. Finely powdered zinc when exposed to the air in moist condition may ignite spontaneously; with sulphur it forms a combustible mixture, not unlike gunpowder. This form should, therefore, be stored and handled with especial care. It should be kept dry and protected from undue contact with the air or contamination with foreign substances, in well-filled, air-tight containers.

I

**ZINC, FREE FROM ARSENIC, SULPHUR,
PHOSPHORUS AND IRON**

This grade is usually in the form of sticks, granules or "mossy" pieces. It dissolves slowly but entirely in dilute sulphuric acid. A piece of platinum foil kept in contact with the zinc hastens the solution of the latter, though it is not itself attacked.

Maximum Limits of Impurities

Arsenic (As)	0.000025%
Matter Oxidizable by Permanganate (as Fe) ...	0.0056%
Compounds of Sulphur and Phosphorus	0.0000%

Methods of Testing

Arsenic.—Introduce 20 gm. of the zinc and 4 gm. of cadmium sulphate into the generating flask of a Marsh apparatus, add 25 to 50 mls of 15% to 20% sulphuric acid, or sufficient to cover the zinc and start a moderate evolution of hydrogen, and allow the action to proceed until the zinc is almost completely dissolved. No deposit of arsenic should be visible in the reduction tube.

Matter Oxidizable by Permanganate (Iron, etc.).—Dissolve 10 gm. of zinc in a mixture of 60 mls of water and 15 mls of sulphuric acid in a flask provided with a rubber valve. No black flocks should remain undissolved. As soon as the zinc has dissolved add to the solution tenth-normal potassium permanganate solution until a distinct pink color is produced. Not more than 0.1 mil should be required.

If more than 0.1 mil of the permanganate solution is used, run a control test, using the same amounts of sulphuric acid and water as before, but no zinc, and correct the titration for the amount required by the solvent alone. Clean platinum foil may be used in this test to accelerate solution of the zinc.

Compounds of Sulphur and Phosphorus.—Into a narrow test-tube introduce 1 gm. of the zinc together with 5 to 10 mls of 15% to 20% sulphuric acid, and, if necessary, a small piece of clean platinum foil, and in the upper part of the tube insert a plug of cotton, which serves to hold back the water carried off by the escaping hydrogen. Cover the mouth of the tube with a small piece of filter paper which has been moistened with 50% silver nitrate solution and carefully dried. Allow the tube to stand two hours in a dark place, perfectly free from hydrogen sulphide. No yellow or black color should develop on the filter paper.

II

ZINC, FREE FROM ARSENIC, NEARLY FREE FROM IRON

This grade of zinc is usually obtainable in granules, sticks, filings, coarse powder and "mossy" pieces. It is slowly soluble in dilute sulphuric acid, frequently leaving a slight residue of black, insoluble floccules.

Maximum Limits of Impurities

Arsenic (As)	0.000025%
Matter Oxidizable by Permanganate (as Fe) ...	0.0280%
Chlorides (Cl)	0.0020%

Methods of Testing

Arsenic.—Perform the test as directed under Zinc, Free from Arsenic, Sulphur, Phosphorus and Iron. The result should be as there indicated.

Matter Oxidizable by Permanganate (Iron, etc.).—Perform this test as directed under Zinc, Free from Arsenic, Sulphur, Phosphorus and Iron. Only a small quantity of black flocks should remain undissolved in the acid mixture. The volume of tenth-normal potassium permanganate solution required to produce a distinct pink color should not exceed 0.5 mil.

Chlorides.—This test is to be applied only to zinc in the form of coarse powder.

Dissolve 5 gm. of the zinc in a mixture of 50 mils of nitric acid and 100 mils of water and add silver nitrate solution. Not more than a slight opalescence should result.

III

ZINC, FREE FROM ARSENIC

This grade of zinc is used in the form of granules, sticks, "mossy" pieces, coarse powder and filings. It usually dissolves readily in dilute sulphuric acid, the rate of speed depending upon the amount of foreign metals present. A zinc, however, that yields an exceedingly brisk evolution of hydrogen is not well adapted to the detection of arsenic by the Marsh test, since traces of arsenic may escape detection by being distributed over the whole surface of the reduction tube, or by being carried even beyond it.

Maximum Limit of Impurity

Arsenic 0.000025%

Method of Testing

Arsenic.—Perform the test as directed under Zinc, Free from Arsenic, Sulphur, Phosphorus and Iron. The result should be as there indicated.

IV

ZINC DUST

Zinc dust is a fine, gray powder, consisting of a mixture of metallic zinc, zinc oxide and usually small quantities of other metals, sulphides, and other volatile constituents of the zinc ore. It contains at least 90% of Zn (metallic). The amount of zinc oxide present depends partly upon the care with which air is excluded in storage. Zinc dust absorbs nitrogen from the air and in moist condition readily occludes oxygen.

Maximum Limit of Impurity

Nitrogen 0.0042%

Methods of Testing

Nitrogen.—Dissolve 20 gm. in a mixture of 20 mls of sulphuric acid and 200 mls of water with the aid of heat. In case the zinc dust dissolves with great difficulty, add a few drops of platinum chloride solution, free from nitric acid. Cool the solution, add 100 mls of 27% sodium hydroxide solution and distil off about 75 mls, collecting the distillate in 10 mls of tenth-normal hydrochloric acid. Titrate the excess acid with tenth-normal alkali solution, using methyl orange as indicator. Not more than 0.6 ml of the tenth-normal acid solution should be neutralized by the ammonia in the distillate.

Quantitative Method.—Weigh out accurately about 0.5 gm. of zinc dust, place it in a beaker, add 50 mls of potassium dichromate solution, containing in each ml 0.04 gm. of $K_2Cr_2O_7$, and then, while stirring well, 10 mls of 10% sulphuric acid in 5 ml portions, and allow the mixture to stand fifteen minutes, stirring frequently. When the zinc has dissolved (a slight insoluble residue usually remains) add 100 mls of water, 5 to 10 mls of concentrated sulphuric acid, and, from a burette, 25 mls of ferrous sulphate solution containing about 200 gm. of ferrous sulphate and 50 mls of sulphuric acid in each liter, and continue adding

the ferrous sulphate solution, about 1 mil at a time, until a drop of the liquid gives a distinct blue color with potassium ferricyanide solution. Then titrate back with the dichromate solution until the reaction with the ferricyanide solution just ceases. In a similar manner ascertain the volume of the dichromate solution which is equivalent to the volume of the ferrous sulphate solution used in the titration. The difference between this equivalent and the amount of dichromate solution used in titrating the zinc should correspond to at least 90% of Zn.

1 mil of empirical $K_2Cr_2O_7$ (equivalent to 0.04 gm. of $K_2Cr_2O_7$) = 0.026663 gm. of Zn, log 42591.

The content of metallic zinc in zinc dust may also be calculated from the volume of hydrogen gas evolved when the zinc dust is dissolved in dilute sulphuric acid. This method is quite generally used in the routine evaluation of zinc dust for technological purposes. A description of the apparatus and procedure are to be found in Scott's Standard Methods of Chemical Analysis, 2d ed., revised, page 487 (1918).

ZINC CHLORIDE

$ZnCl_2$

Mol. Wt. 136.29

Zinc chloride usually occurs as white granules or powder, sometimes in fused pieces or sticks. The salt is very deliquescent. It dissolves in less than 1 part of water and is also readily soluble in alcohol. Much water decomposes it, forming the insoluble oxychloride.

Zinc chloride cannot be fully dehydrated by heat. When heated above 250° C. it fuses and at higher temperatures sublimes. If too high a temperature is maintained during fusion, considerable basic chloride is likely to form which will interfere with the solubility of the cooled melt. The salt is seldom entirely free from oxychloride.

Zinc chloride is used principally as a dehydrating agent; as a solvent for cellulose; as a reagent for resorcinol and

alkaloids; and in reagents for the detection of cellulose, silk and mercerized cotton.

The reagent should be kept in well-stoppered bottles and exposed to the air as little as possible. Zinc chloride is very caustic and should not be allowed to remain in contact with the skin.

Maximum Limits of Impurities

Zinc Oxychloride (as ZnO)	2.5000%
Suphates (SO ₃)	0.0050%
Foreign Heavy Metals	0.0000%
Alkali Salts	0.2000%

Methods of Testing

Zinc Oxychloride.—Dissolve 1 gm. in 1 mil of well-boiled water. The solution should not be more than slightly turbid. Then add 3 mils of alcohol and 0.05 mil of hydrochloric acid. The resulting liquid should be clear, any turbidity existing in the aqueous solution or produced by the alcohol disappearing upon the addition of the acid.

Sulphates.—Dissolve 1 gm. in 10 mils of water, acidulate with a few drops of hydrochloric acid and add barium chloride solution. No turbidity should be produced.

Foreign Heavy Metals.—Dissolve 1 gm. in 10 mils of water, add 1 mil of hydrochloric acid and some hydrogen sulphide water. No change should be apparent.

Dissolve 1 gm. in 10 mils of water and add 10 mils of ammonia water. The solution should be colorless. Then add 5 drops of hydrogen sulphide water. A pure white precipitate should form.

Alkali Salts.—Dissolve 1 gm. in 10 mils of water, add 10 mils of ammonia water, dilute to 100 mils and pass in hydrogen sulphide gas until the zinc is completely precipitated. Filter, evaporate the filtrate to dryness and ignite and weigh the residue. It should not weigh more than 0.002 gm.

ZINC IODIDE-STARCH SOLUTION

(Trommsdorff's Reagent)

Zinc iodide-starch solution is a colorless liquid, clear or sometimes slightly opalescent. It may be prepared by heating 4 parts of starch, 20 parts of zinc chloride and 100 parts of water for an hour or more at or near the boiling point, replacing from time to time the water lost by evaporation. The resulting solution is diluted to 1 liter and, after clarification by filtration or sedimentation, 2 parts of zinc iodide are added.

Zinc iodide-starch solution is used mainly for the detection of nitrites, free chlorine and other oxidizing agents, and as an indicator.

The solution spoils on exposure to air and light, acquiring a blue color. In this condition it is unfit for use. It should therefore be kept in well-stoppered bottles, protected from light.

Method of Testing

Mix 1 mil of the solution with 20 mils of water and add 1 drop of tenth-normal iodine solution. A deep blue color should be produced.

Mix 1 mil of the zinc iodide-starch solution with 50 mils of water and add diluted sulphuric acid. A blue color should not develop.

ZINC OXIDE

ZnO

Mol. Wt. 81.37

Zinc oxide is usually a white or yellowish-white, amorphous powder, insoluble in water, but soluble in dilute acetic or mineral acids and in ammonia water and ammonium carbonate solution. Exposed to the air, zinc oxide gradually absorbs carbon dioxide which may be expelled by ignition. When heated, it takes on a yellow color and on cooling again assumes its original appearance.

Zinc oxide is chiefly used in the titration of manganese by the Volhard method; as a neutralizing agent, for ex-

ample in the estimation of phosphates by titration with silver nitrate solution; for converting sulphocyanates to cyanates; and for precipitating iron and aluminum.

The reagent should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Substances Insoluble in Sulphuric Acid	0.0000%
Arsenic (As)	0.0015%
Sulphates (SO_4)	0.0050%
Chlorides (Cl)	0.0025%
Nitrates (N_2O_5)	0.0016%
Carbonates	0.0000%
Calcium (Ca)	0.0200%
Magnesium (Mg)	0.0050%
Foreign Heavy Metals	0.0000%
Substances Reducing Permanganate	0.0000%

Methods of Testing

Substances Insoluble in Sulphuric Acid (Lead).—Dissolve 5 gm. in 100 mls of 10% sulphuric acid. The solution should be clear and no insoluble residue should remain.

Arsenic.—Mix 1 gm. with 3 mls of stannous chloride solution and allow it to stand one hour. A dark color should not develop.

Sulphates.—Shake 2 gm. with 20 mls of water, filter and to the filtrate add a few drops of hydrochloric acid and some barium chloride solution. No change should be apparent.

Chlorides.—Shake 2 gm. with 20 mls of water, filter and to the filtrate add a few drops of nitric acid and some silver nitrate solution. Not more than a slight opalescence should result.

Nitrates.—Dissolve 1 gm. in 10 mls of 36% acetic acid, add 1 drop of indigo solution and a few milligrams of sodium chloride and mix the liquid with 10 mls of sulphuric acid. The blue color should persist after the mixing.

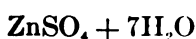
Carbonates.—Dissolve the zinc oxide in diluted acetic or mineral acid. No effervescence should be perceptible.

Calcium and Magnesium.—Dissolve 1 gm. in 10 mls of 36% acetic acid, add 20 mls of ammonia water and then some ammonium oxalate and ammonium phosphate solutions. No change should be apparent after the addition of either reagent.

Foreign Heavy Metals.—Dissolve 1 gm. in 10 mls of 36% acetic acid and add 20 mls of ammonia water and 5 drops of hydrogen sulphide water. A pure white precipitate should be produced.

Substances Reducing Permanganate.—Very carefully triturate 3 gm. of zinc oxide in a mortar with 20 mls of water containing in solution 0.2 gm. of ferric ammonium sulphate (free from ferrous salt). Add to the mixture 40 mls of 10% sulphuric acid and effect complete solution by gently heating. Dilute the solution with 100 mls of water which has just been thoroughly boiled and then cooled, and then add 0.05 ml of tenth-normal potassium permanganate solution. A distinct pink color should be imparted to the liquid.

ZINC SULPHATE



Mol. Wt. 287.54

Zinc sulphate is usually in the form of colorless crystals, efflorescent in dry air. It is soluble in about 0.6 part of water but is almost insoluble in alcohol. Its aqueous solution is acid to litmus.

The salt is used principally in precipitating proteoses and in standardizing sodium sulphide solution employed in the volumetric determination of zinc. It should be stored in well-stoppered bottles.

Maximum Limits of Impurities

Chlorides (Cl)	0.0010%
Nitrates (N_2O_5)	0.0016%
Iron (Fe)	0.0010%

Other Foreign Metals	0.0000%
Ammonium Salts (NH ₃)	0.0035%
Arsenic (As)	0.0005%

Methods of Testing

Chlorides.—Dissolve 1 gm. in 20 mls of water and add silver nitrate solution. No change should be apparent.

Nitrates.—Dissolve 1 gm. in 10 mls of water, add a few milligrams of sodium chloride, one drop of indigo solution and finally 10 mls of sulphuric acid. The blue color should not disappear on shaking the mixture.

Iron.—Dissolve 1 gm. in 20 mls of water, add a few drops of nitric acid, boil and then add potassium sulphocyanate solution. The mixture should not be reddened.

Other Foreign Metals.—Dissolve 0.5 gm. in a mixture of 10 mls of water and 5 mls of ammonia water. The solution should be clear and colorless. Now add 5 drops of hydrogen sulphide water. The precipitate should be pure white.

Ammonium Salts.—Boil 1 gm. of zinc sulphate with 10 mls of 27% sodium hydroxide solution. The vapors should not affect moist red litmus paper.

Arsenic.—Dissolve 2 gm. in 20 mls of water and introduce the solution in small quantities gradually into a Marsh apparatus. No deposit of arsenic should be obtained after the action has proceeded for one hour.

ZINC SULPHIDE, NEUTRAL

For Soil Analysis

ZnS

Mol. Wt. 97.43

Neutral zinc sulphide for soil analysis is a yellowish or grayish powder, insoluble in water but soluble in dilute mineral acids with copious evolution of hydrogen sulphide.

This reagent is used chiefly in conjunction with neutral calcium chloride for determining the acidity of soils. (See the reference to this method under Calcium Chloride, Neu-

tral.) For this purpose it should be free from basic compounds.

Exposed to the air zinc sulphide gradually decomposes with evolution of hydrogen sulphide; therefore the reagent should be stored in well-stoppered bottles.

Method of Testing

Test for Basicity.—Put 0.1 gm. in 100 mls of neutral water and add 2 drops of methyl orange solution. The addition of 0.2 ml of tenth-normal acid solution should produce a distinct pink color, which should not disappear on shaking the mixture for at least one minute.

INTERNATIONAL ATOMIC WEIGHTS

	Symbol.	Atomic Weight.		Symbol.	Atomic Weight.
Aluminum	Al	27.1	Neodymium	Nd	144.3
Antimony	Sb	120.2	Neon	Ne	20.2
Argon	A	39.88	Nickel	Ni	58.68
Arsenic	As	74.96	Niton (radium emanation)	Nt	222.4
Barium	Ba	137.37	Nitrogen	N	14.01
Bismuth	Bi	208.0	Osmium	Os	190.9
Boron	B	11.0	Oxygen	O	16.00
Bromine	Br	79.92	Palladium	Pd	106.7
Cadmium	Cd	112.40	Phosphorus	P	31.04
Cæsium	Cs	132.81	Platinum	Pt	195.2
Calcium	Ca	40.07	Potassium	K	39.10
Carbon	C	12.005	Praseodymium	Pr	140.9
Cerium	Ce	140.25	Radium	Ra	226.0
Chlorine	Cl	35.46	Rhodium	Rh	102.9
Chromium	Cr	52.0	Rubidium	Rb	85.45
Cobalt	Co	58.97	Ruthenium	Ru	101.7
Columbium	Cb	93.1	Samarium	Sa	150.4
Copper	Cu	63.57	Scandium	Sc	44.1
Dysprosium	Dy	162.5	Selenium	Se	79.2
Erbium	Er	167.7	Silicon	Si	28.3
Europium	Eu	152.0	Silver	Ag	107.88
Fluorine	F	19.0	Sodium	Na	23.00
Gadolinium	Gd	157.3	Strontium	Sr	87.63
Gallium	Ga	69.9	Sulphur	S	32.06
Germanium	Ge	72.5	Tantalum	Ta	181.5
Glucium	Gl	9.1	Tellurium	Te	127.5
Gold	Au	197.2	Terbium	Tb	159.2
Helium	He	4.00	Thallium	Tl	204.0
Holmium	Ho	163.5	Thorium	Th	232.4
Hydrogen	H	1.008	Thulium	Tm	168.5
Indium	In	114.8	Tin	Sn	118.7
Iodine	I	126.92	Titanium	Ti	48.1
Iridium	Ir	193.1	Tungsten	W	184.0
Iron	Fe	55.84	Uranium	U	238.2
Krypton	Kr	82.92	Vanadium	V	51.0
Lanthanum	La	139.0	Xenon	Xe	130.2
Lead	Pb	207.20	Ytterbium (Neoytterbium)	Yb	173.5
Lithium	Li	6.94	Yttrium	Yt	88.7
Lutecium	Lu	175.0	Zinc	Zn	65.37
Magnesium	Mg	24.32	Zirconium	Zr	90.6
Manganese	Mn	54.93			
Mercury	Hg	200.6			
Molybdenum	Mo	96.0			

ACETIC ANHYDRIDE

Calculation of percentages of acetic anhydride and acetic acid in mixtures of the two from the total acidity of the mixture, expressed as acetic acid, obtained by titration with standard alkali solution.*

Total Acidity as HC ₂ H ₃ O ₂ Per Cent.	Equivalent to a Mix- ture of		Total Acidity as HC ₂ H ₃ O ₂ Per Cent.	Equivalent to a Mix- ture of	
	(CH ₃ CO) ₂ O Per Cent.	HC ₂ H ₃ O ₂ Per Cent.		(CH ₃ CO) ₂ O Per Cent.	HC ₂ H ₃ O ₂ Per Cent.
108.82	50	50	.55	77	23
109.00	51	49	.73	78	22
.19	52	48	.90	79	21
.37	53	47	114.10	80	20
.52	54	46	.28	81	19
.70	55	45	.47	82	18
.88	56	44	.61	83	17
110.08	57	43	.80	84	16
.94	58	42	.98	85	15
.40	59	41	115.17	86	14
.58	60	40	.35	87	13
.77	61	39	.52	88	12
.95	62	38	.71	89	11
111.12	63	37	.90	90	10
.28	64	36	116.10	91	9
.45	65	35	.27	92	8
.62	66	34	.46	93	7
.80	67	33	.65	94	6
.98	68	32	.82	95	5
112.14	69	31	117.02	96	4
.32	70	30	.20	97	3
.45	71	29	.36	98	2
.60	72	28	.50	99	1
.80	73	27	117.65	100	0
113.00	74	26			
.23	75	25			
.38	76	24			

* Worden, Technology of Cellulose Esters, VIII, 2911 (1916).

By permission of Dr. E. C. Worden.

ACETIC ACID

Congealing points of concentrated acetic acid*

Percentage of Acetic Acid.	Congealing Point.	Percentage of Acetic Acid.	Congealing Point.	Percentage of Acetic Acid.	Congealing Point.
100.00	16.63°	99.45	15.57°	98.80	14.32°
99.95	16.53	99.40	15.48	98.70	14.13
99.90	16.43	99.35	15.38	98.60	13.94
99.85	16.34	99.30	15.29	98.55	13.75
99.80	16.24	99.25	15.19	98.40	13.56
99.75	16.15	99.20	15.10	98.30	13.37
99.70	16.05	99.15	15.00	98.20	13.17
99.65	15.96	99.10	14.91	98.10	12.98
99.60	15.86	99.05	14.81	98.00	12.79
99.55	15.77	99.00	14.71		
99.50	15.67	98.90	14.52		

* Worden, Technology of Cellulose Esters, VIII, 2908 (1916).

By permission of Dr. E. C. Worden.

HYDROCHLORIC ACID

Percentages of HCl corresponding to specific gravities of hydrochloric acid at 60°/60° F.*

Sp. Gr. 60°/60° F.	Per Cent. HCl.	Sp. Gr. 60°/60° F.	Per Cent. HCl.	Sp. Gr. 60°/60° F.	Per Cent. HCl.
1.0069	1.40	1.0943	18.82	1.1563	30.71
1.0140	2.82	1.0985	19.63	1.1590	31.27
1.0211	4.25	1.1027	20.45	1.1619	31.82
1.0284	5.69	1.1069	21.27	1.1647	32.38
1.0357	7.15	1.1111	22.09	1.1675	32.93
1.0394	7.89	1.1154	22.92	1.1703	33.50
1.0432	8.64	1.1197	23.75	1.1732	34.07
1.0469	9.40	1.1240	24.57	1.1760	34.64
1.0507	10.17	1.1265	25.06	1.1789	35.21
1.0545	10.94	1.1292	25.56	1.1817	35.78
1.0584	11.71	1.1319	26.05	1.1846	36.35
1.0623	12.48	1.1345	26.56	1.1875	36.93
1.0662	13.26	1.1372	27.07	1.1904	37.58
1.0701	14.04	1.1399	27.58	1.1934	38.26
1.0741	14.83	1.1426	28.09	1.1963	38.95
1.0781	15.62	1.1453	28.61	1.1993	39.64
1.0821	16.41	1.1480	29.13	1.2023	40.32
1.0861	17.21	1.1508	29.65	1.2053	41.01
1.0902	18.01	1.1535	30.18		

* Compiled from the table by W. C. Ferguson, J. Soc. Chem. Ind., 24, 789 (1905).

NITRIC ACID

Percentages of HNO_3 corresponding to specific gravities of nitric acid at $60^\circ/60^\circ \text{ F.}^*$

Sp. Gr. $60^\circ/60^\circ \text{ F.}$	Per Cent. HNO_3	Sp. Gr. $60^\circ/60^\circ \text{ F.}$	Per Cent. HNO_3	Sp. Gr. $60^\circ/60^\circ \text{ F.}$	Per Cent. HNO_3
1.0741	12.86	1.1885	30.49	1.3303	52.30
1.0781	13.49	1.1934	31.21	1.3364	53.32
1.0821	14.13	1.1983	31.94	1.3426	54.36
1.0861	14.76	1.2033	32.68	1.3488	55.43
1.0902	15.41	1.2083	33.42	1.3551	56.52
1.0943	16.05	1.2134	34.17	1.3615	57.65
1.0985	16.72	1.2185	34.94	1.3679	58.82
1.1027	17.38	1.2236	35.70	1.3744	60.06
1.1069	18.04	1.2288	36.48	1.3810	61.38
1.1111	18.70	1.2340	37.26	1.3876	62.77
1.1154	19.36	1.2393	38.06	1.3942	64.20
1.1197	20.02	1.2446	38.85	1.4010	65.67
1.1240	20.69	1.2500	39.66	1.4078	67.18
1.1284	21.36	1.2554	40.47	1.4146	68.73
1.1328	22.04	1.2609	41.30	1.4216	70.33
1.1373	22.74	1.2664	42.14	1.4286	71.98
1.1417	23.42	1.2719	43.00	1.4356	73.67
1.1462	24.11	1.2775	43.89	1.4428	75.40
1.1508	24.82	1.2832	44.78	1.4500	77.17
1.1554	25.53	1.2889	45.68	1.4573	79.03
1.1600	26.24	1.2946	46.58	1.4646	81.08
1.1647	26.96	1.3004	47.49	1.4721	83.33
1.1694	27.67	1.3063	48.42	1.4796	85.70
1.1741	28.36	1.3122	49.35	1.4872	88.32
1.1789	29.07	1.3182	50.32	1.4948	91.35
1.1837	29.78	1.3242	51.30	1.5026	95.11

* Compiled from the table by W. C. Ferguson, J. Soc. Chem. Ind., **24**, 788 (1905).

SULPHURIC ACID

Percentages of H_2SO_4 corresponding to specific gravities of sulphuric acid at 60°/60° F.*

Sp. Gr. 60°/60° F.	Per Cent. H_2SO_4 .	Sp. Gr. 60°/60° F.	Per Cent. H_2SO_4 .	Sp. Gr. 60°/60° F.	Per Cent. H_2SO_4 .
1.0000	0.00	1.2288	30.79	1.5934	68.13
1.0069	1.02	1.2393	32.05	1.6111	69.65
1.0140	2.08	1.2500	33.33	1.6292	71.17
1.0211	3.13	1.2609	34.63	1.6477	72.75
1.0284	4.21	1.2719	35.93	1.6667	74.36
1.0357	5.28	1.2832	37.26	1.6860	75.99
1.0432	6.37	1.2946	38.58	1.7059	77.67
1.0507	7.45	1.3063	39.92	1.7262	79.43
1.0584	8.55	1.3182	41.27	1.7470	81.30
1.0662	9.66	1.3303	42.63	1.7683	83.34
1.0741	10.77	1.3426	43.99	1.7901	85.66
1.0821	11.89	1.3551	45.35	1.7957	86.33
1.0902	13.01	1.3679	46.72	1.8012	87.04
1.0985	14.13	1.3810	48.10	1.8068	87.81
1.1069	15.25	1.3942	49.47	1.8125	88.65
1.1154	16.38	1.4078	50.87	1.8182	89.55
1.1240	17.53	1.4216	52.26	1.8239	90.60
1.1328	18.71	1.4356	53.66	1.8297	91.80
1.1417	19.89	1.4500	55.07	1.8354	93.19
1.1508	21.07	1.4646	56.48	1.8381	94.00
1.1600	22.25	1.4796	57.90	1.8407	95.00
1.1694	23.43	1.4948	59.32	1.8427	96.00
1.1789	24.61	1.5104	60.75	1.8437	97.00
1.1885	25.81	1.5263	62.18	1.8439	97.50
1.1983	27.03	1.5426	63.66	1.8437	98.00
1.2083	28.28	1.5591	65.13	1.8424	99.00
1.2185	29.53	1.5761	66.63	1.8391	100.00

* Compiled from the table by W. C. Ferguson, J. Soc. Chem. Ind., 24, 790 (1905), and from the table for sulphuric acid from 94% to 100% H_2SO_4 by H. B. Bishop, Van Nostrand's Chemical Annual, 1918. Prof. J. C. Olsen Editor, by permission of D. Van Nostrand Company, Publishers, New York.

FUMING SULPHURIC ACID

The composition of fuming sulphuric acid expressed in percentages of H_2SO_4 and free SO_3 and the corresponding percentages of total SO_3 .*

Actual Composition.		Total SO_3 . Per Cent.	Actual Composition.		Total SO_3 . Per Cent.
Free SO_3 . Per Cent.	H_2SO_4 . Per Cent.		Free SO_3 . Per Cent.	H_2SO_4 . Per Cent.	
0	100	81.63	25	75	86.22
1	99	81.82	26	74	86.41
2	98	82.00	27	73	86.59
3	97	82.18	28	72	86.78
4	96	82.37	29	71	86.96
5	95	82.55	30	70	87.14
6	94	82.73	31	69	87.33
7	93	82.92	32	68	87.51
8	92	83.10	33	67	87.69
9	91	83.29	34	66	87.88
10	90	83.47	35	65	88.06
11	89	83.65	36	64	88.24
12	88	83.84	37	63	88.43
13	87	84.02	38	62	88.61
14	86	84.20	39	61	88.80
15	85	84.39	40	60	88.98
16	84	84.57	41	59	89.16
17	83	84.75	42	58	89.35
18	82	84.94	43	57	89.53
19	81	85.12	44	56	89.71
20	80	85.31	45	55	89.90
21	79	85.49	46	54	90.08
22	78	85.67	47	53	90.27
23	77	85.86	48	52	90.45
24	76	86.04	49	51	90.63

* Compiled from the table by H. B. Bishop, Van Nostrand's Chemical Annual, 1918. Prof. J. C. Olsen Editor, by permission of D. Van Nostrand Company, Publishers, New York.

AMMONIA WATER

Percentages of NH_3 corresponding to specific gravities of ammonia water at 60°/60° F.*

Sp. Gr. 60°/60° F.	Per Cent. NH_3 .	Sp. Gr. 60°/60° F.	Per Cent. NH_3 .	Sp. Gr. 60°/60° F.	Per Cent. NH_3 .
1.0000	0.00	0.9396	15.84	0.9003	28.42
0.9947	1.21	0.9365	16.80	0.8989	28.91
0.9894	2.46	0.9333	17.76	0.8974	29.40
0.9842	3.73	0.9302	18.72	0.8960	29.89
0.9790	5.02	0.9272	19.68	0.8946	30.38
0.9739	6.31	0.9241	20.64	0.8931	30.87
0.9689	7.61	0.9211	21.60	0.8917	31.36
0.9655	8.49	0.9180	22.56	0.8903	31.85
0.9622	9.38	0.9150	23.52	0.8889	32.34
0.9589	10.28	0.9121	24.50	0.8875	32.83
0.9556	11.18	0.9091	25.48	0.8861	33.32
0.9524	12.10	0.9061	26.46	0.8847	33.81
0.9492	13.02	0.9047	26.95	0.8833	34.30
0.9459	13.96	0.9032	27.44	0.8819	34.79
0.9428	14.90	0.9018	27.93		

* Compiled from the table by W. C. Ferguson, J. Soc. Chem. Ind., 24, 787 (1905).

METHYL ALCOHOL

Specific gravities at 15°/15° C. of mixtures of methyl alcohol and water and the corresponding percentages (by volume) of methyl alcohol.*

Per Cent. CH ₃ OH by Volume.	Sp. Gr. 15°/15° C.	Per Cent. CH ₃ OH by Volume.	Sp. Gr. 15°/15° C.	Per Cent. CH ₃ OH by Volume.	Sp. Gr. 15°/15° C.
0	1.00000	34	.95708	68	.89790
1	.99851	35	.95576	69	.89561
2	.99703	36	.95443	70	.89327
3	.99560	37	.95308	71	.89088
4	.99422	38	.95170	72	.88844
5	.99283	39	.95029	73	.88596
6	.99146	40	.94886	74	.88346
7	.99011	41	.94741	75	.88092
8	.98877	42	.94593	76	.87836
9	.98746	43	.94443	77	.87578
10	.98621	44	.94291	78	.87312
11	.98496	45	.94136	79	.87040
12	.98370	46	.93979	80	.86760
13	.98247	47	.93820	81	.86474
14	.98125	48	.93657	82	.86180
15	.98003	49	.93493	83	.85883
16	.97884	50	.93326	84	.85582
17	.97766	51	.93155	85	.85276
18	.97648	52	.92982	86	.84967
19	.97530	53	.92806	87	.84646
20	.97413	54	.92626	88	.84314
21	.97295	55	.92443	89	.83971
22	.97177	56	.92256	90	.83623
23	.97058	57	.92067	91	.83269
24	.96939	58	.91877	92	.82907
25	.96820	59	.91682	93	.82538
26	.96700	60	.91483	94	.82163
27	.96580	61	.91282	95	.81772
28	.96459	62	.91079	96	.81363
29	.96338	63	.90873	97	.80942
30	.96216	64	.90663	98	.80514
31	.96091	65	.90450	99	.80082
32	.95966	66	.90234	100	.79647
33	.95838	67	.90014		

* Compiled from the table published in U. S. Bureau of Standards Circular No. 19 (1911).

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